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Test Protocol Supplemental Fuels Test Burn in Marquette Cement Kiln at Oglesby, Illinois

Systech Corp.
Xenia, OH

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TEST PROTOCOL
SUPPLEMENTAL FUELS TEST BURN IN MARQUETTE CEMENT KILN AT OGLESBY, ILLINOIS

EPA Contract Number 68-01-6071

Randolph Chrismon

U.S. Environmental Protection Agency
Resource Recovery Division (WH-563)
401 M Street, SW
Washington, DC 20460

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This test protocol was developed to evaluate the technical and environmental consequences of burning flammable liquid wastes as a supplemental fuel in a dry process cement kiln. Primary objectives were to determine the kiln destruction and removal efficiency (DRE) of the principle organic hazardous constituents (POHCs) in the liquid waste, to evaluate the impact of burning the waste upon atmospheric emissions and waste kiln dust with special emphasis on trace metals and chlorine, and to observe the impact on the quality of the cement clinker produced.

This protocol presents the experimental approach and the sampling, analytical, and quality assurance procedures required to achieve these objectives.

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DISCLAIMER

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FOREWORD

Significant quantities of Hi-Btu hazardous liquid wastes are currently being landfilled at considerable expense. One attractive alternative to landfill disposal is the utilization of these high energy wastes as supplemental fuels in the cement manufacturing industry. The USEPA/RRD has continued to seek environmentally sound applications of waste resources in industry. SYSTECH Corporation, through an ongoing research contract (EPA Contract No. 68-01-6071, Task No. 20) with the USEPA/RRD, was asked to develop a test protocol which would explore the technical and environmental consequences of burning flammable liquid wastes in a dry process cement kiln.

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LIST OF ABBREVIATIONS

AA	--atomic absorption
CC	--configuration control
CFR	--Code of Federal Regulations
DRE	--destruction and removal efficiency
dscf	--dry standard cubic feet
dscfm	--dry standard cubic feet per minute
EP	--extraction procedure
EPA	--Environmental Protection Agency
ESP	--electrostatic precipitator
FID	--flame ionization detector
GC	--gas chromatograph
M	--EPA Method
NDIR	--nondispersive infrared
POHC	--principal organic hazard constituent
ppm	--parts per million
QC	--quality control
TGNMO	--total gaseous nonmethane organics
TPD	--tons per day

SECTION 1

INTRODUCTION

OBJECTIVES

The objectives of this testing are to demonstrate the utilization of select combustible liquid wastes as supplemental fuel in the Portland cement manufacturing process, to achieve a reduction in the fossil fuel consumption of the kiln process, and to effectively destroy the waste materials. Although the practice is currently being applied at several cement kilns in the United States, no detailed testing has been performed on those facilities achieving energy recovery by firing principally nonchlorinated, high Btu wastes. The majority of tests have been on kilns used as disposal mechanisms and have been performed using highly chlorinated wastes in kilns located outside of the United States. The subject testing will investigate the use of wastes selected for their fuel values, typically consisting of ink and paint manufacturing wastes and solvent refiner still bottoms which have 2 to 5 percent levels of chlorine but contain concentrations of metals resulting from pigments in the paint and ink wastes. Major attention will be given in this investigation to the emission levels of metals from the process and to the destruction and removal efficiency (DRE) of the kiln on the principal organic hazardous constituents (POHCs) of the liquid waste.

PLANT DESCRIPTION

The kiln to be tested in this demonstration is owned by the Marquette Company. It is not currently burning waste fuels and will be specially equipped for the testing. The kiln is a dry process kiln with an

electrostatic precipitator (ESP) system for emission controls. It is fired using a blend of coal and petroleum coke. The cement plant is capable of producing approximately 1,400 tons per day (TPD) of cement clinker and, at a 15 percent substitution rate, would fire approximately 10,000 gal/day of waste solvent fuels.

The kiln testing will be relatively brief, consisting of 2 days of baseline testing under normal operating conditions using solid fuels and a 2-day test burn during which approximately 20,000 gal of liquid fuels will be fired. A major aspect of the testing will be to investigate stack emissions from the kiln. Both baseline and test conditions will include measurements of metals in the particulate discharged from the stack; total gaseous nonmethane organics (TGNMO); selected POHCs; carbon monoxide, oxygen, and carbon dioxide; and particulate, sulfur dioxide, nitrogen oxide, and chlorine emissions. The liquid fuels will be analyzed for composition including trace metals, PCBs, organic constituents, water, chlorine, sulfur, nitrogen, and ash. Attempts will be made to correlate fuel composition with any changes observed in the stack emissions occurring during the baseline condition versus the liquid fuel feed condition. The coal will be analyzed to identify any variations in quality during the two test conditions. The kiln feed will be sampled and retained for future reference. The cement clinker product will be tested for composition and mechanical characteristics using standard industry techniques with special attention given to the trace metals and chlorine quantified in the liquid fuel. Ash from the ESP will be analyzed for sulfur, chlorine, and trace metals and will be subjected to an Extraction Procedure (EP) toxicity test.

A schematic showing the major cement kiln components and material flows is presented in Figure 1. A schematic of the waste fuels test piping system which will be used to introduce solvent fuel into the cement kiln is shown in Figure 2.

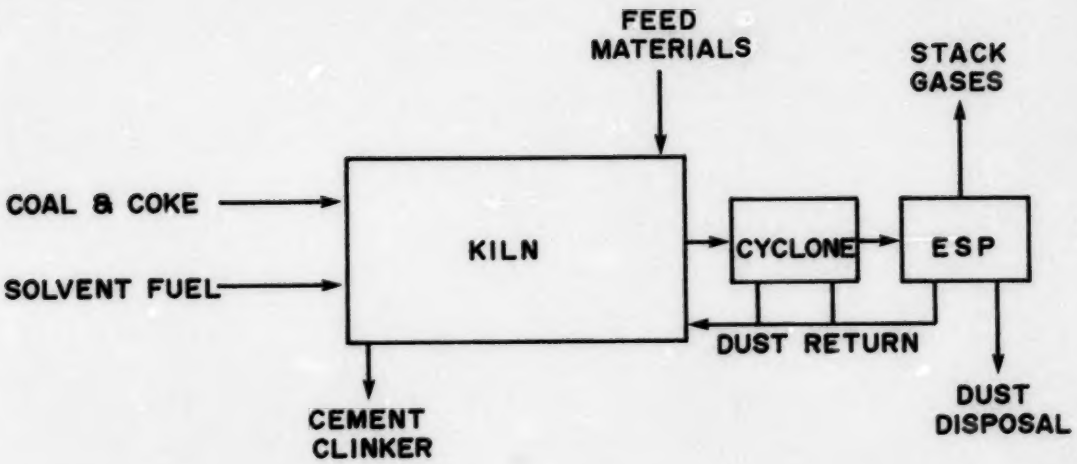


Figure 1. Marquette-Oglesby cement kiln schematic.

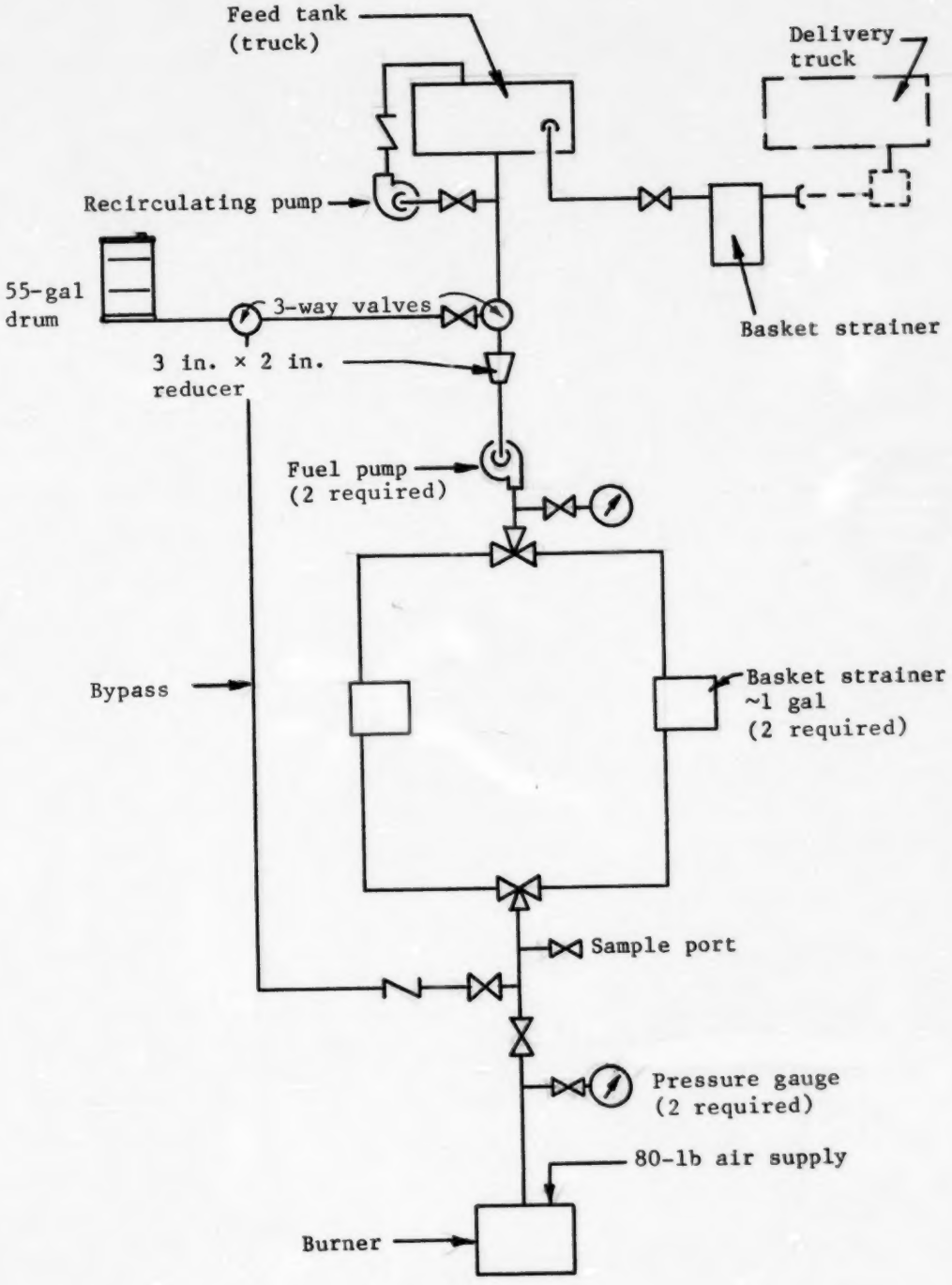


Figure 2. Waste fuels test piping schematic.

SAMPLE SITE LOCATIONS

All process streams except the combustion air will be sampled during this demonstration. The fuels, waste dust, stack gases, and kiln feed and product will be sampled as follows.

Fuels

Liquid Fuel--

The liquid fuel samples will be taken from a T-valve installed in the liquid fuel delivery system illustrated in Figure 2. The sample valve will be located in the recirculating portion of the system between the basket strainers and the delivery nozzle.

Solid Fuel--

The coal and coke fuel mixture will be sampled at the end of the conveyor just after the belt scale (illustrated in Figure 3). At this location it will be possible to obtain a full cross section sample of the belt contents just prior to entering the kiln.

ESP Dust

Samples of waste dust from the last two banks of the ESP will be obtained at the end of the screw conveyor leading from the ESP (illustrated in Figure 4). Dust from the first three banks is recycled into the kiln.

Stack Gases

Samples of the stack gases will be obtained from the ports installed 55 ft above the base of the stack as illustrated in Figure 5. Oxygen, carbon monoxide, and carbon dioxide will be monitored continuously by analyzing a sample stream taken from one of the two central ports. The other central port will be available for TGNMO, NO_x, and POHCs sampling. The two outside ports are at 90 degrees to each other. They will be reserved for particulate,

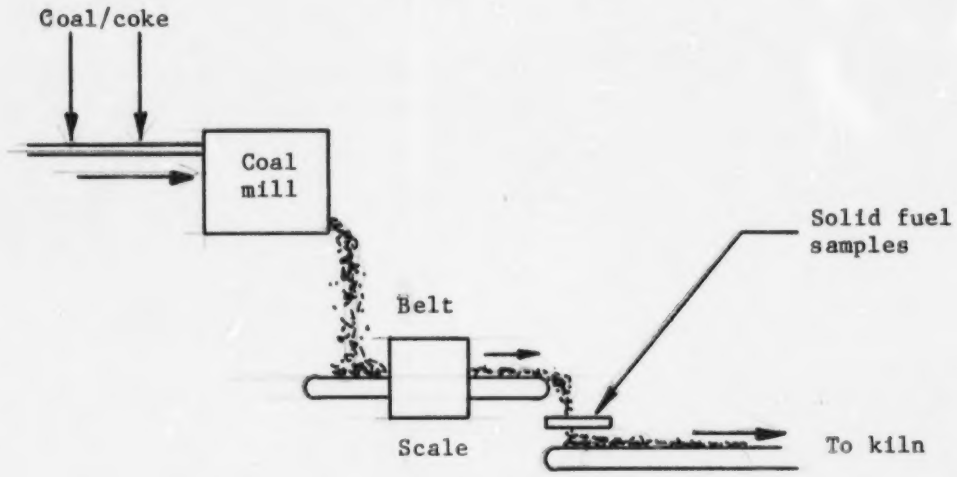


Figure 3. Solid fuel sampling location.

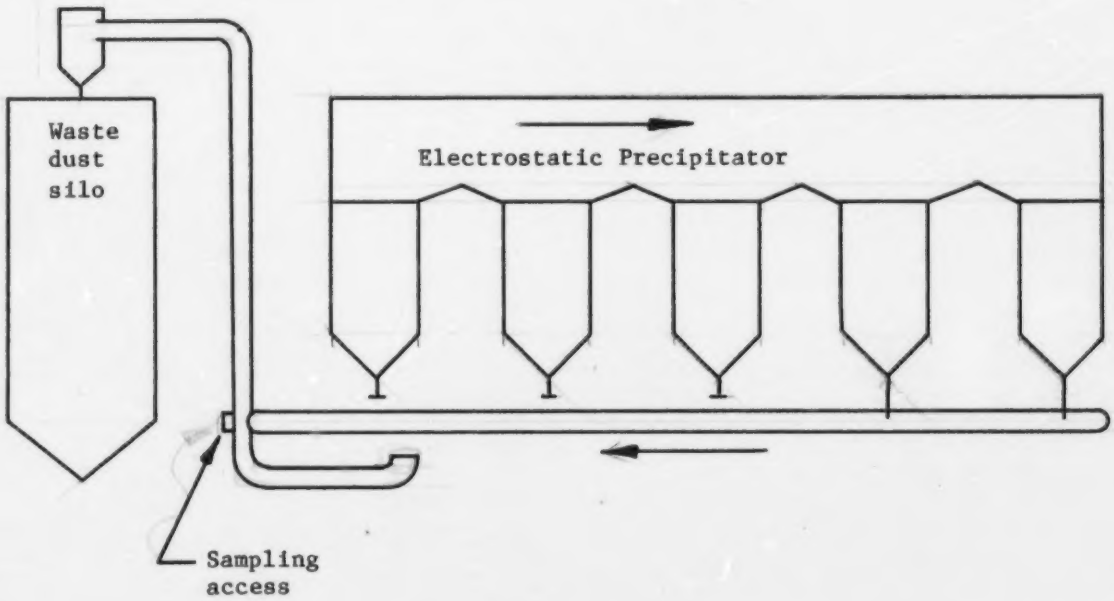


Figure 4. Waste dust sampling location.

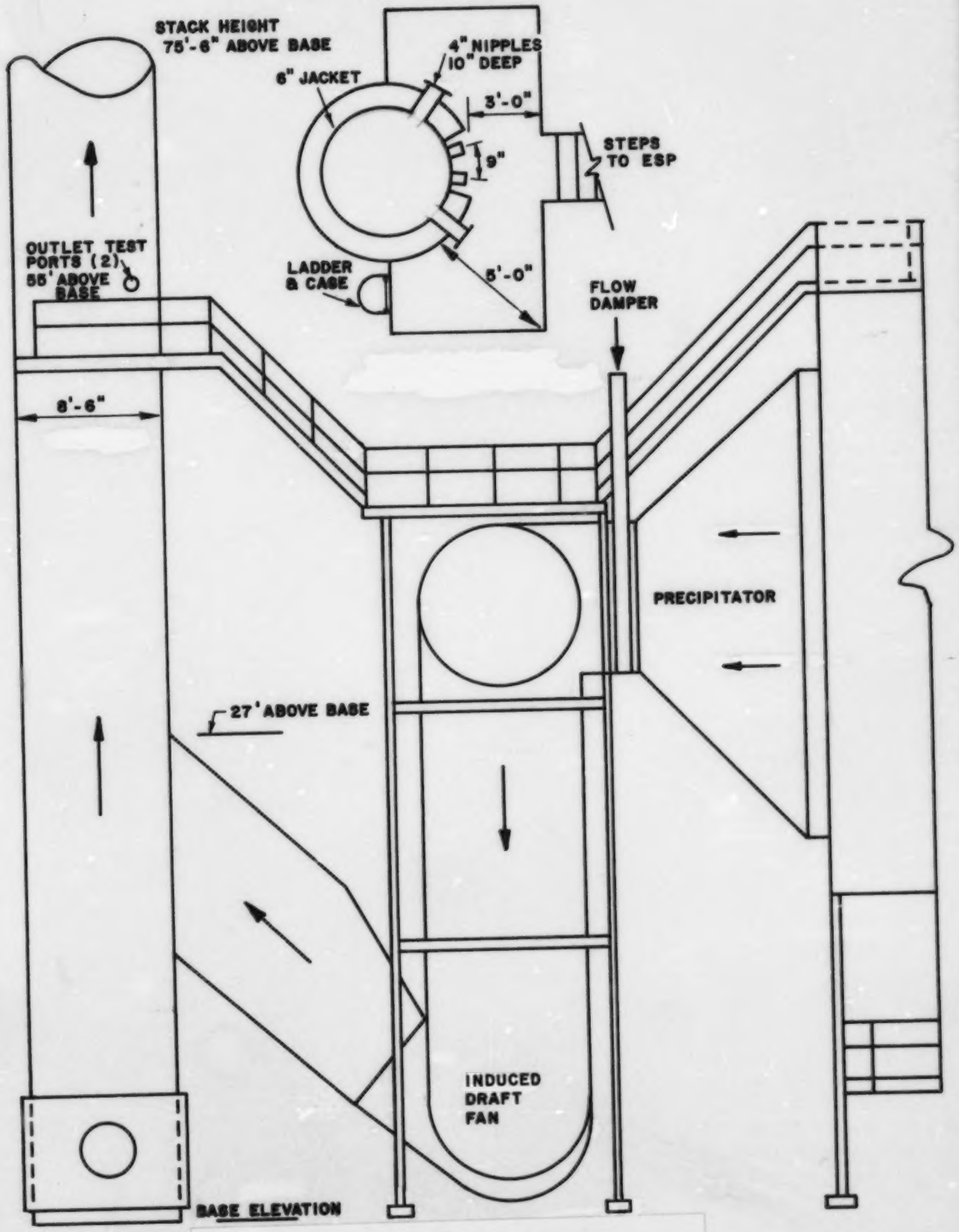
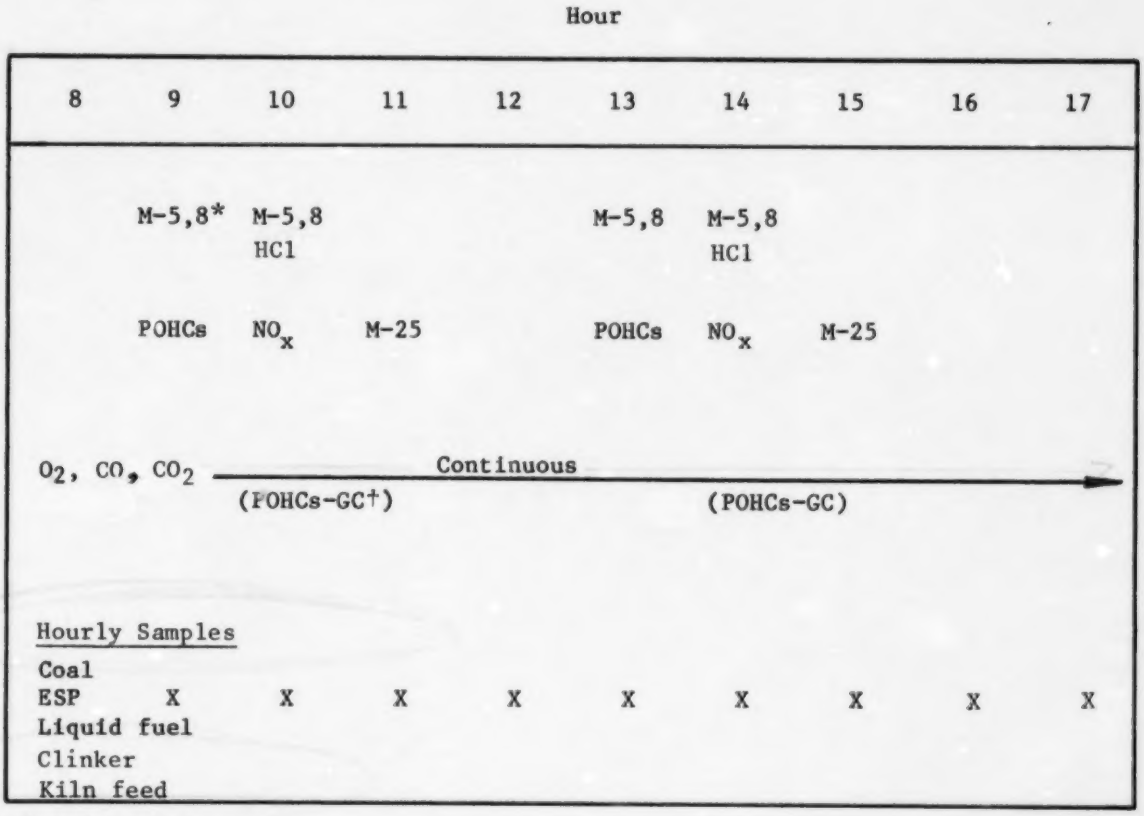


Figure 5. Stack sampling locations.

SO_2, and HCl sampling. The approximate timing sequence of these tests is illustrated in Figure 6, the daily test schedule.

Kiln Feed and Clinker

The kiln feed and clinker will be sampled in the same manner and at the same locations as they are during routine production runs.



* EPA Method
† gas chromatograph

Figure 6. Daily test schedule.

SECTION 2

STACK EMISSION CHARACTERIZATION

Stack emissions will be sampled and analyzed for 2 days during normal process operations (baseline) and for 2 days of co-firing coal and liquid fuel. The test parameters will include the following: particulate concentration and trace metals content of the collected particulate; O_2, CO_2, CO, SO_2, and NO_x gas concentrations; hydrochloric acid emissions; and total gaseous nonmethane organics concentrations. Of special interest will be the monitoring of the stack for the presence of POHC species. Table 1 summarizes the methods to be used in the acquisition of this data, and the remainder of this section details specific procedures that are not standard methodologies or which represent modifications of standard methodologies.

PARTICULATE AND SO_2 EMISSIONS

Particulate emissions and SO_2 will be determined twice each day at the stack by using a modification of the standard procedures for particulates and SO_2 referenced in the Code of Federal Regulations (CFR), Title 40, Part 60, Appendix A, Methods 1 through 5 and Method 8. The modification of these standard procedures consists of substituting 3 percent H_2O_2 for distilled water in the first two impingers of the Method 5 sampling train. This modification will have no impact on the particulate determination but will measure sulfuric acid mist and SO_2 together rather than separately as in the Method 8 procedure. The SO_3 will be collected by the H_2O_2 and measured as SO_2. Although this will increase the apparent SO_2 emissions, the SO_3 emissions are normally very low compared with the total SO_x emissions. The

TABLE 1. STACK SAMPLING TEST MATRIX

Parameter measured	Sampling method	Sample frequency	Number samples	Sample size	Analytical method	Reporting Units
STACK						
Stack dimensions	EPA M-1	once	-	-	-	inches
Gas flow rate	EPA M-2	2/day	-	-	-	dscfm*
Gas temperature	EPA M-2	2/day	-	-	-	°F
Gas composition	EPA M-3	2/day	8	5ℓ	EPA M-3	percent
Gas moisture content	EPA M-4	2/day	8	40 ft^3	EPA M-4	percent
Particulate concentration	EPA M-5	2/day	8	40 ft^3	EPA M-5	lb/hr
Particulate trace metals	EPA M-5	2/day	8	40 ft^3	AA	lb/hr
SO_2 concentration	EPA M-5,8†	2/day	8	40 ft^3	EPA M-8	lb/hr
NO_x concentration	EPA M-7	6/day	24	2ℓ	EPA M-7	lb/hr
TGNMO	EPA M-25	2/day	8	5ℓ	EPA M-25 (GC/FID)§	mg/hr
HCl	EPA draft procedure	2/day	8	40ℓ	EPA draft	lb/hr
CO	EPA M-10	Continuous	-	-	EPA M-10 (NDIR)‡	ppm**
CO_2	EPA M-10	Continuous	-	40ℓ	NDIR	percent
O_2	EPA M-10	Continuous	-	-	Electrochemical cell	percent
POHCs	EPA M-23	2/day	8	100ℓ	EPA M-23 (GC/FID)	lb/hr

* dry standard cubic feet per minute
† modified
§ flame ionization detector
‡ nondispersive infrared
** parts per million

large diameter of the stack will require the use of a stainless steel probe to avoid excess breakage problems which would result from the use of a Pyrex, glass lined probe normally required for the Method 8 procedure.

PARTICULATE TRACE METALS

The particulate matter recovered from the probe acetone rinsings and the filters of the Method 5 trains will be combined and analyzed by atomic absorption (AA) spectroscopy for the following trace metals: zinc, chromium, copper, lead, manganese, nickel, mercury, and cadmium. It should be noted that nickel and chromium values may be elevated by the use of the stainless steel probe in the Method 5 sampling train. Also note that the majority of the mercury may be in a vapor state until it passes from the stack, and it will not be associated with the particulate.

OXYGEN, CARBON MONOXIDE, AND CARBON DIOXIDE

The oxygen, carbon monoxide, and carbon dioxide levels in the stack gas will be monitored continuously during the test periods with instrumentation located in SYSTECH's mobile laboratory. A sample of the gas stream will be pumped continuously through heated Teflon tubing to the laboratory trailer at the base of the stack. This gas stream will be conditioned by removing particulate matter and water vapor at the stack. Particulate matter will be removed with a sintered metal filter located in the stack, and water vapor will be condensed from the gas stream by passing the sample through an iced condensing chamber located just outside the stack.

Carbon monoxide will be analyzed according to the specifications of the CFR, Title 40, Part 60, Appendix A, Method 10, Determination of Carbon Monoxide Emission from Stationary Sources, using a Beckman Model 864 nondispersive infrared analyzer as the analytical instrument. This instrument meets all procedural specifications except that it does not have a linear response. To compensate for this nonlinearity, a 4-point calibration will be performed twice daily to ensure consistent instrument response.

The carbon dioxide content of the gas stream will also be monitored with a Beckman Model 864 NDIR analyzer. This instrument will be calibrated twice each day with a 4-point calibration.

The oxygen content of the gas stream will be monitored on a Theta Sensor Source Monitor, Series 7213, Model 1940. The principle of operation of this instrument is based on the combined use of a semipermeable membrane and selective oxidation-reduction reactions within completely sealed electrochemical transducers. The electrical signal generated by this transducer is directly proportional to the concentration of oxygen in the gas stream. The Theta Sensor oxygen instrument will be calibrated twice daily using zero grade nitrogen and a 10 percent O_2 standard.

The electrical response of both instruments will be recorded on a Leeds and Northrup Speedo-Max W multipoint strip chart recorder. These data will be analyzed and the average values for each 4-hr test period reported.

NITROGEN OXIDES

The concentration of nitrogen oxide in the stack will be determined using the procedures specified in the CFR, Title 40, Part 60, Appendix A, Method 7. NO_x concentrations will be determined twice each day by taking grab samples at the stack. As indicated in Method 7, each data point will consist of three grab samples which will be analyzed individually and reported as an average.

TOTAL GASEOUS NONMETHANE ORGANICS

Total gaseous nonmethane organics will be determined at the stack twice each day by following the procedures specified in the Code of Federal Regulations, Title 40, Part 60, Appendix A, Method 25. The grab samples taken on site will be stored in dry ice until they can be analyzed by the appropriate GC technique.

HYDROCHLORIC ACID EMISSIONS

The sampling and analytical procedure to be employed for determination of hydrochloric acid emissions is an Environmental Protection Agency (EPA) draft procedure. This procedure calls for the use of a midget impinger sampling train with dilute sodium hydroxide as the capture media and a mercuric nitrate titration as the analytical methodology. Hydrochloric acid emissions will be sampled twice each day at the stack.

PRINCIPAL ORGANIC HAZARDOUS CONSTITUENTS

The POHCs chosen for analysis in this test are methylene chloride, 1,1,1-trichloroethane; methyl ethyl ketone; and toluene. Methods of fuel sampling and analysis for these constituents are described in Section 3 of this document. Stack sampling for the POHCs will be conducted twice each day (simultaneously with the Method 5 tests) during the coal baseline and liquid fuel test burns, by the proposed EPA Method 23 (see Federal Register, June 11, 1980, and EPA 600/4-80-003). Sample analysis will be performed by the GC/FID technique described in this method. The sensitivity of the GC will be adjusted to provide a response to 0.1 ppm of the POHCs. This sensitivity will permit the calculation of POHC destruction and removal efficiencies of 99.5 to 99.99 percent, depending on the complexity of the sample matrix and the amount of POHCs present.

SECTION 3

FUEL, DUST, AND PRODUCT CHARACTERIZATION

Coal and liquid fuel will be sampled on an hourly basis to verify the consistency of the coal and to quantify liquid fuel characteristics and POHCs. Dust from the cyclones and the first three banks of the electrostatic precipitator is recycled back into the process while dust from the last two banks of the ESP is considered to be a process waste stream. This waste dust will be sampled hourly to determine if components of the liquid fuel are reporting to this stream and, if so, if there is any reason for environmental concern. Samples of the clinker will be taken hourly to verify that process control parameters and product quality are being maintained within acceptable limits. Kiln feed will be sampled hourly, and the samples will be retained for future reference. The test matrix to be employed for the coal, liquid fuel, ESP dust, and clinker is shown in Table 2. One quarter of each of the hourly grab samples from the above streams will be composited into 4-hr analytical samples which will correspond with stack emissions test periods. The remaining three quarters of each hourly sample will be maintained as a record sample for future reference.

COAL

The feed rate of the coal will be monitored hourly by observing the readout of the belt scale which is located downstream of the coal mill. Coal samples will be taken hourly from the conveyor just after the belt scale. These hourly grab samples will be composited into a 4-hr analytical sample representing the coal quality during each 4-hr test period. These composited coal samples will be analyzed by Marquette's Laboratory utilizing their routine coal analysis procedures.

TABLE 2. PROCESS SAMPLING TEST MATRIX

Parameter measured	Sampling method	Sample frequency	Number samples	Sample size	Analytical Method	Reporting Units
LIQUID FUEL						
Feed rate	Observation	Hourly	-	-	Monitoring truck level and calibrated pressure gauge	lb/hr
Heating value	Grab sample from T-valve in feed line	Hourly, composited to two 4-hr analytical samples/day	8	1ℓ	ASTM-D240-76	Btu/lb
Chloride					ASTM-D808-63	Percent wt.
Sulfur					ASTM-D129-64	Percent wt.
Nitrogen					ASTM-E258-67	Percent wt.
Percent water					ASTM-D1744-64	Percent vol.
Ash					ASTM-D482-63	Percent wt.
Specific gravity					Gravimetric	lb/gal
Viscosity					Viscometer	Centipoise
Solids					Centrifugation	Percent vol.
Organic constituents >0.1%					GC/FID	Percent wt.
PCB's >50 ppm					GC/EC	ppm
Trace metals					AA	ppm
POHCs					GC/FID	lb/hr
ESP DUST						
Collection rate	Empty dust silo grab sample from dust conveyor	1/day	-	-	Truck weight	Ton/hr
Sulfur		Hourly, composited to two 4-hr analytical samples/day	8	1ℓ	$BaSO_4$ precipitation	Percent wt.
Chlorides					$Hg(NO_3)_2$ titration	Percent wt.
Trace metals					AA	mg/kg
EP toxicity					EP Tox/AA	mg/ℓ
CLINKER						
Marquette process control parameters	Marquette	Hourly	-	-	Marquette	
Trace metals	Marquette	Hourly, composited to two 4-hr analytical samples/day	8	1ℓ	AA	mg/kg
Chlorides					$Hg(NO_3)_2$ titration	Percent wt.
CEMENT						
Strength	-	-	-	-	ASTM C-109	Percent change between baseline and liquid fuel cement properties
Expansion	-	-	-	-	ASTM C-452	
Flow	-	-	-	-	ASTM C-230	
Air content	-	-	-	-	ASTM C-185	
Fineness	-	-	-	-	ASTM C-184	
Autoclave exposure	-	-	-	-	ASTM C-151	
Gilmore setting time	-	-	-	-	ASTM C-266	
Portland cement spec.	-	-	-	-	ASTM C-150-80	

LIQUID FUEL

The feed rate of the liquid fuel will be determined by comparing the results of two different methods: (1) the volume of fuel remaining in the truck will be calculated hourly by measuring the liquid level and correlating it with the truck geometry and (2) the real-time feed rate will be determined by observing the nozzle pressure hourly. The feed rates at various nozzle pressures will be determined prior to testing by feeding from a 55-gal drum (illustrated in Figure 2) and by observing the time required to empty it.

Samples of the liquid fuel will be taken hourly during the test period from the feed line. Aliquots of these hourly samples will be composited into samples representative of the 4-hr test periods and analyzed for the parameters listed in Table 2.

ELECTROSTATIC PRECIPITATOR DUST

Dust collected from the last two banks of the ESP is considered a solid waste stream and is disposed. The accumulation rate of this ESP dust will be monitored by emptying the dust silo at the beginning of a test sequence and allowing dust to accumulate for a 24-hr period. The accumulated dust will then be emptied into a truck which will be weighed. Grab samples of the ESP dust will be collected hourly from the end of the dust conveyor just prior to being picked up by the screw conveyor system which transfers it to the dust silo. These hourly samples will be composited into 4-hr analytical samples. These samples will be analyzed for sulfur and chloride content and trace metals (including those metals regulated by EP toxicity). Sulfur and chloride determinations will be made by bomb calorimeter techniques using benzoic acid as an accelerator to ensure complete combustion. A sample of the dust will also be extracted with hot water to determine the water soluble chlorides and sulfur content. The dust will be analyzed for the same trace metals as the liquid fuel by standard AA spectroscopy techniques. The leachability of the

trace metals in the waste dust will be determined by procedures called out in the CFR, Title 40, Part 261, Appendix II, Section 7, Extraction Procedure Toxicity.

CLINKER AND CEMENT QUALITY

Mineral composition and component ratios of the clinker and physical and mechanical characteristics of the cement are product quality and process control parameters which are normally monitored by Marquette on a routine basis. These will be sampled and analyzed on an hourly basis by Marquette using their standard procedures throughout the liquid fuel test period. In addition, portions of the hourly clinker samples will be composited into 4-hr samples and analyzed for water soluble chlorides, total chlorine, and trace metals content.

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APPENDIX A-1

DATA SHEETS AND CALCULATION SETS

METHOD 5

PARTICULATE FIELD DATA

PLANT _____ AMBIENT TEMPERATURE _____ NOZZLE DIAMETER, in. _____ STACK NO. _____

DATE _____ BAROMETRIC PRESSURE _____ STACK DIAMETER, in. _____ RUN NO. _____

LOCATION _____ ASSUMED MOISTURE, % _____ METER ΔH @ _____ FUEL _____

OPERATOR _____ PROBE LENGTH, in. _____ C FACTOR _____ BOILER SIZE _____

SAMPLING TIME/POINT _____ LEAK CHECK _____ PROCESS WEIGHT RATE _____

TIME	TRAVERSE POINT		VELOCITY HEAD Δ Ps	STATIC PRESSURE (IN. H_2O)	PUMP VACUUM IN Hg GUAGE	PRESSURE DIFFERENTIAL ACROSS ORIFICE METER (ΔH) IN., H_2O ACTUAL	DRY GAS SAMPLE VOL. (Vm) FT^3	TEMPERATURES °F					
START /STOP	NO.	LOCATION IN.						STACK (T_s)	PROBE	OVEN	IMP OUT	IMP IN	METER OUT (Tm)

ORSAT MEASUREMENT

	TIME	CO_2	O_2	CO	N_2
1					
2					
3					
4					
5					
6					

COMMENTS:

_____

_____

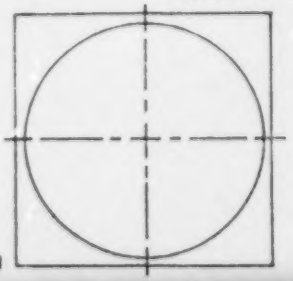
_____

_____

_____

_____

SCHEMATIC OF STACK



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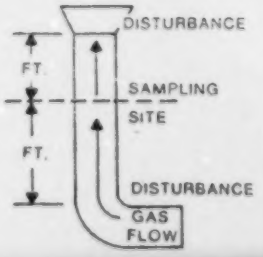

SAMPLE RECOVERY AND INTEGRITY DATA FORM

Plant _____ Sample date _____

Sample location _____ Run number _____

Sample recovery person _____ Recovery date _____

Filter(s) number _____

MOISTURE

Impingers		Silica gel		
Final volume (wt)	_____ml (g)	Final wt	_____g	_____g
Initial volume (wt)	_____ml (g)	Initial wt	_____g	_____g
Net volume (wt)	_____ml (g)	Net wt	_____g	_____g

Total moisture _____g

Color of silica gel _____

Description of impinger water _____

RECOVERED SAMPLE

Blank filter container number _____ Sealed _____

Filter container number _____ Sealed _____

Description of particulate on filter _____

Acetone rinse container number _____ Liquid level marked? _____

Acetone blank container number _____ Liquid level marked? _____

Samples stored and locked _____

Remarks _____

_____

Date of laboratory custody _____

Laboratory personnel taking custody _____

Remarks _____

_____

METHOD 5 PARTICULATE EMISSION CALCULATIONS

Test No. _____ Time _____ To _____ Date _____ Project _____

Analyst _____

Supervisor _____

1. VOLUME METERED AT STANDARD CONDITIONS

$$V_{m(std)} = 17.64\ V_m Y\ \frac{P_{bar} + \frac{\Delta H}{13.6}}{T_m}$$

$$17.64\ (\quad)(\quad)\ \frac{(\quad) + \frac{(\quad)}{13.6}}{(\quad)} = ____\ \text{dscf}$$

Where: $V_{m(std)}$ = dry gas volume measured by the dry gas meter, corrected to standard conditions, dscf

V_m = dry gas volume measured by dry gas meter, dcf

Y = dry gas mater calibration factor

P_{bar} = barometric pressure, inches Hg

ΔH = average pressure differential across the orifice meter, inches H_2O

T_m = absolute average dry gas meter temperature, °R

Test No. _____
Project No. _____

2. VOLUME OF WATER VAPOR

$V_{w(std)} = 0.04707\ (V_{lc})$

$0.04707\ (\quad) = _____$ scf

Where: $V_{w(std)}$ = volume of water vapor in the gas sample corrected to standard conditions, scf

V_{lc} = total volume of liquid collected in impingers and silica gel

3. MOISTURE CONTENT

$$B_{ws} = \frac{V_{w(std)}}{V_{m(std)} + V_{w(std)}}$$

$$\frac{(\quad)}{(\quad) + (\quad)} = _____$$

Where: B_{ws} = water vapor in the gas stream proportion by volume

$V_{w(std)}$ = volume of water vapor in the gas sample corrected to standard conditions, scf

$V_{m(std)}$ = volume of gas sampled measured by the dry gas meter corrected to standard conditions, dscf

Test No. _____
Project No. _____

4. CROSS SECTIONAL AREA OF STACK OR DUCT

Circular duct:

$$A = 5.454 \times 10^{-3} (d)^2$$

$$5.454 \times 10^{-3} (\quad\quad) = _____ \text{ ft}^2$$

Rectangular duct:

$$A = (L \times W)\ 6.944 \times 10^{-3}$$

$$(\quad\quad)(\quad\quad)\ 6.944 \times 10^{-3} = _____ \text{ ft}^2$$

Where: A = cross sectional area of duct, ft^2

L = length of rectangular duct, inches

W = width of rectangular duct, inches

d = diameter of circular duct, inches

5. MOLECULAR WEIGHT OF STACK GAS

$$M_d = 0.440\ (\%\ CO_2) + 0.320\ (\%\ O_2) + 0.280\ (\%\ N_2 + \%\ CO)$$

$$0.440\ (\quad) + 0.320\ (\quad) + 0.280\ (\quad\quad) = _____ \text{lb/lb mole}$$

$$M_s = M_d\ (1 - B_{ws}) + 18.0\ B_{ws}$$

$$(\quad)(1 - \quad) + 18\ (\quad) = _____ \text{ lb/lb mole}$$

Where: M_d = molecular weight of stack gas, dry basis, lb/lb mole

M_s = molecular weight of stack gas, wet basis, lb/lb mole

B_{ws} = water vapor in the gas stream, proportion by volume

Test No. _____
Project No. _____

6. AVERAGE STACK GAS VELOCITY

$$V_s = 85.49\ C_p \sqrt{(\Delta P)_{ave}} \sqrt{\frac{T_{s(ave)}}{P_s\ M_s}}$$

$$85.49\ (\quad)\ \sqrt{(\qquad)}\ \sqrt{\frac{\qquad\qquad}{(\quad)(\quad)}} = _____\ \text{ft/sec}$$

Where: v_s = average stack gas velocity, ft/sec

C_p = pitot tube coefficient, dimensionless

ΔP = velocity pressure in stack, inches H_2O

T_s = absolute stack temperature, °R

P_s = absolute stack gas pressure, inches Hg

M_s = molecular weight of stack gas, wet basis, lb/lb mole

Test No. _____
Project No. _____

7. AVERAGE STACK GAS DRY VOLUMETRIC FLOW RATE, STANDARD CONDITIONS

$$Q_{std} = 3{,}600\ (1 - B_{ws})\ V_s\ A\ \frac{T_{std}}{T_{s(ave)}}\ \frac{P_s}{P_{(std)}}$$

3,600 (1-)( )( ) ——— ——— = _____ dscf/hr

Where: Q_{std} = dry volumetric stack gas flow rate corrected to standard conditions, dscf/hr

B_{ws} = water vapor in the gas stream, proportion by volume

V_s = average stack gas velocity, ft/sec

A = cross sectional area of duct, ft^2

T_{std} = standard absolute temperature, 528°R

T_s = absolute stack temperature, °R

P_s = absolute stack gas pressure, inches Hg

P_{std} = standard absolute pressure, 29.92 inches Hg

Test No. _____
Project No. _____

8. AVERAGE STACK GAS DRY VOLUMETRIC FLOW FATE, ACTUAL CONDITIONS

$$Q_{act} = Q_{std} \frac{\frac{T_{s(ave)}}{T_{std}} \frac{P_s}{P_{std}}}{1 - B_{ws}}$$

$$(\quad) \frac{\frac{_____}{_____}}{1 - _____} = _____ \text{ acf/hr}$$

Where: Q_{act} = actual stack gas volumetric flow rate, acf/hr

B_{ws} = water vapor in the gas stream, proportion by volume

V_s = average stack gas velocity, ft/sec

P_s = absolute stack gas pressure, inches Hg

T_s = absolute stack temperature, °R

T_{std} = standard absolute temperature, 528°R

P_{std} = standard absolute pressure, 29.92 in Hg

Test No. _____
Project No. _____

9. ISOKINETIC VARIATION

$$I = 0.09450 \frac{T_s V_{m(std)}}{P_s V_s A_n \Theta (1 - B_{ws})}$$

$$0.09450 \frac{(\quad)(\quad)}{(\quad)(\quad)(\quad)(\quad)(1 - \quad)} = _____ \text{ percent}$$

Where:

I = percent of isokinetic sampling

T_s = absolute temperature of stack, °R

$V_{m(std)}$ = volume of gas sample measured by the dry gas meter, corrected to standard conditions, dscf

P_s = absolute stack gas pressure, inches Hg

V_s = stack gas velocity

A_n = cross sectional area of nozzle

Θ = sampling time, minutes

B_{ws} = water vapor in gas stream, proportion by volume

Test No. _____
Project No. _____

10. PARTICULATE CONCENTRATION

$$C_s = 2.205 \times 10^{-6} \frac{M_n}{V_{m(std)}}$$

$$2.205 \times 10^{-6} \quad \frac{\qquad}{\qquad} = _____ \text{ lb/scf}$$

Where: C_s = concentration of stack gas particulates, dry

M_n = total amount of particulate collected, mg

$V_{m(std)}$ = volume of gas sampled measured by the dry gas meter corrected to standard conditions, dscf

11. PARTICULATE EMISSION RATE

$$E = (C_s)\ (Q_{std})$$

( ) ( ) = _____ lb/hr

Where: E = particulate emission rate, lb/hr

C_s = concentration of stack gas particulate, dry basis corrected to standard conditions, lb/dscf

Q_{std} = dry volumetric stack gas flow rate corrected to standard conditions, dscf/hr

ALLOWABLE EMISSIONS

The allowable emissions for this source are calculated from Rule 203 of the State of Illinois Pollution Control Board Regulations, June 1976.

The appropriate portion of this rule used is "Standards for Existing Emission Sources" which states that for process weights in excess of 30 ton/hr, the following formula shall be used:

$$E = [55 (P^{0.11})] - 40$$

where

E = allowable emission in lb/hr

P = process weight in ton/hr

For this test sequence, P is equal to the combined feed rates of the following inputs:

P = coal/coke + liquid fuel + kiln feed

APPENDIX A-2

DATA SHEETS AND CALCULATION SETS

METHOD 25

TGNMO FIELD DATA

P/N _____ RUN NUMBER _____

FACILITY _____ DATE _____

LOCATION _____ OPERATOR _____

PROCESS _____ REGULATOR SETTING _____ cc/min

REGULATOR NO. _____ STACK TEMPERATURE _____ °F

TANK NUMBER(s) _____ TRAP NUMBER (s) _____

	Pretest	Post Test
BAROMETRIC PRESS., mm Hg		
TANK VACUUM mm Hg		
GAUGE VACUUM "Hg		
LEAK CHECK mm/5 min.		
AMBIENT TEMPERATURE °C		

SAMPLE TIME 24 hr clock/minutes	GAUGE VACUUM inches Hg	COMMENTS

T.G.N.M.O. DATA SHEET

Trap No. _____ Tank No. _____

SAMPLE NO. _____ DATE TRAP BURNOUT/ANALYST _____ / _____

CLIENT _____ DATE TRAP ANALYSIS/ANALYST _____ / _____

TEST NO. _____ DATE TANK ANALYSIS/ANALYST _____ / _____

FIELD DATA

PRE-TEST BAROMETRIC PRESSURE	$P_{B(1)}$	_____	mm Hg	POST-TEST BAROMETRIC PRESSURE	$P_{B(2)}$	_____	mm Hg
PRE-TEST TANK VACUUM	$P_{T(1)}$	_____	mm Hg	POST-TEST TANK VACUUM	$P_{T(2)}$	_____	mm Hg
PRE-TEST TEMPERATURE	T_1	_____	°C	POST-TEST TEMPERATURE	T_2	_____	°C

TANK PRESSURIZATION DATA

LAB BAROMETRIC PRESSURE	$P_{B(3)}$	_____	mm Hg	TANK VOLUME	V_T	_____	L
LAB TEMPERATURE	T_3	_____	°C	FINAL TANK PRESSURE	$P_{T(3)}$	_____	mm Hg

TRAP BURNOUT

RUN A

COLLECTION VESSEL NO.		_____		COLLECTION VESSEL VOLUME	V_{CV}	_____	L
LAB BAROMETRIC PRESSURE	$P_{B(4)}$	_____	mm Hg	INITIAL VACUUM	$P_{CV(1)}$	_____	mm Hg
LAB TEMPERATURE	T_4	_____	°C	FINAL PRESSURE	$P_{CV(2)}$	_____	mm Hg

RUN B

COLLECTION VESSEL NO.		_____		COLLECTION VESSEL VOLUME	V_{CV}	_____	L
LAB BAROMETRIC PRESSURE	$P_{B(4)}$	_____	mm Hg	INITIAL VACUUM	$P_{CV(1)}$	_____	mm Hg
LAB TEMPERATURE	T_4	_____	°C	FINAL PRESSURE	$P_{CV(2)}$	_____	mm Hg

RUN C

COLLECTION VESSEL NO.		_____		COLLECTION VESSEL VOLUME	V_{CV}	_____	L
LAB BAROMETRIC PRESSURE	$P_{B(4)}$	_____	mm Hg	INITIAL VACUUM	$P_{CV(1)}$	_____	mm Hg
LAB TEMPERATURE	T_4	_____	°C	FINAL PRESSURE	$P_{CV(2)}$	_____	mm Hg

MEASURED TANK CONCENTRATIONS (as PPM CH_4 , NOT DILUTION CORRECTED) _____, _____, _____. Average _____ $C_{VOC(1)}$

MEASURED COLLECTION VESSEL CONCENTRATIONS (as PPM CH_4, NOT DILUTION CORRECTED)

Run A: _____, _____, _____. Average _____ $C_{VOC(4)}$

Run B: _____, _____, _____. Average _____ $C_{VOC(4)}$

Run C: _____, _____, _____. Average _____ $C_{VOC(4)}$

T.G.N.M.O. CALCULATION SHEET

(ALL DATA CORRECTED TO 293.15°K [68°F, 20°C] AND ONE ATMOSPHERE PRESSURE: [760 mm Hg])

$$\left[\text{CORRECTION FACTOR: } \frac{293.15°K}{760 \text{ mm Hg}} = 0.3857\right]$$

A. SAMPLE VOLUME

$$V_S = (0.3857)(V_T)\left[\frac{(P_{B(2)} - P_{T(2)})}{(T_2 + 273.2)} - \frac{(P_{B(1)} - P_{T(1)})}{(T_1 + 273.2)}\right]$$

B. TRAP CONCENTRATION (DILUTION CORRECTION):

$$C_{VOC(5)} = \left[\frac{(V_{CV})(P_{CV(2)} + P_{B(4)})}{(V_S)(T_4 + 273.2)}\right](C_{VOC(4)})(0.3857)$$

C. TANK CONCENTRATION (DILUTION CORRECTION):

$$C_{VOC(2)} = \frac{\left[\frac{P_{B(3)} + P_{T(3)}}{T_3 + 273.2}\right]}{\left[\frac{(P_{B(2)} - P_{T(2)})}{(T_2 + 273.2)} - \frac{(P_{B(1)} - P_{T(1)})}{(T_1 + 273.2)}\right]}(C_{VOC(1)})$$

D. CONVERSION TO MG/L

Molar Gas Volume at 20°C = 24.055 L/mol

$$MG/L = \left[C_{VOC(2)} + C_{VOC(5)}\right]\left(\frac{12.011}{(24.055)(1000)}\right)$$

CALCULATIONS BASED ON PROCEDURE GIVEN IN FEDERAL REGISTER, OCTOBER 5, 1979.

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T.G.N.M.O. CALCULATION SHEET SYMBOL BREAKDOWN

V_S	Volume of sample (liters)
V_T	Volume of tank (liters)
$P_B(2)$	Post-test barometric pressure (m m Hg)
$P_T(2)$	Post-test tank vacuum (m m Hg)
T_2	Post-test temperature (°C)
$P_B(1)$	Pre-test barometric pressure (m m Hg)
$P_T(1)$	Pre-test tank vacuum (m m Hg)
T_1	Pre-test temperature (°C)
$C_{VOC}(5)$	Dilution corrected trap concentration (PPM CH_4)
V_{CV}	Volume of collection vessel (liters)
$P_{CV}(2)$	Final pressure of collection vessel (m m Hg)
$P_B(4)$	Lab barometric pressure for trap analysis (m m Hg)
T_4	Lab temperature for trap analysis (°C)
$C_{VOC}(4)$	Non-dilution corrected trap concentration (PPM CH_4)
$P_B(3)$	Lab barometric pressure for tank analysis (m m Hg)
$P_T(3)$	Final tank pressure (m m Hg)
T_3	Lab temperature for tank analysis (°C)
$C_{VOC}(2)$	Dilution corrected tank concentration (PPM CH_4)
$C_{VOC}(1)$	Non-dilution corrected tank concentration (PPM CH_4)

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EMISSIONS RATE OF TGNMO

$$E_{TGNMO}\ mg/hr = C_{TGNMO} \times Q_{std}\ 28.32 \ell/ft^3$$

where

E_{TGNMO} = TGNMO emissions rate (mg/hr)

C_{TGNMO} = Concentration of TGNMO in stack gases in carbon equivalents (mg/ℓ)

Q_{std} = Dry volumetric stack gas flow rate corrected to standard conditions (dry standard cubic feet/hr)

APPENDIX A-3

DATA SHEETS AND CALCULATION SETS

METHOD 7

Systech Corporation
245 North Valley Road
Xenia, Ohio 45385
(513) 372-8077
Dayton Number: (513) 429-2533

Field Sampling Data Form for EPA Method 7 -
Determination of NO_2 Emissions from Stationary Sources

Operator _____ Location City _____
Project Number _____ Date _____
Plant Name _____ Sampling Height Above Ground (Feet) _____

	Flask No.	Sample No.	V_f (mℓ)	Sample time	T_I (°F)	$P_{F(I)}$ (mm Hg)	$P_{B(I)}$ (in. Hg)	Recovery time/ date	T_F (°F)	$P_{F(F)}$ (mm Hg)	$P_{B(F)}$ (in. Hg)
1											
2											
3											
4											
5											
6											
7											
8											
9											
10											
11											
12											
13											
14											
15											
16											
17											
18											
19											
20											
21											
22											
23											
24											
25											

Systech Corporation
245 North Valley Road
Xenia, Ohio 45385
(513) 372-8077
Dayton Number: (513) 429-2533

Analytical Data Form for EPA Method 7 - Determination of NO_2 Emissions from Stationary Sources

Analyst _____ Date _____

Project Number _____ Optimum Wavelength (nm) _____

	Sample No.	Sample Volume (ml)	Aliquot Size (ml)	Aliquot Factor	Dilution Factor	Absorbance (OD)
1	Blank	50.0	25.0	2	25/x =	
2	A1 - 100 µg NO_2	50.0	25.0	2	25/x =	
3	A2 - 200 µg NO_2	50.0	25.0	2	25/x =	
4	A3 - 300 µg NO_2	50.0	25.0	2	25/x =	
5	A4 - 400 µg NO_2	50.0	25.0	2	25/x =	
6						
7						
8						
9						
10						
11						
12						
13						
14						
15						
16						
17						
18						
19						
20						

Was Page omitted

Systech Corporation
245 North Valley Road
Xenia, Ohio 45385
(513) 372-8077
Dayton Number: (513) 429-2533

EPA Method 7 - NO_2 Computer Worksheet

Client _____ Location _____ SYSTECH P.N. _____

	Sample No.	V_f (mℓ)	$P_{B(I)}$ (in. Hg)	$P_{B(F)}$ (in. Hg)	$P_{F(I)}$ (mm Hg)	$P_{F(F)}$ (mm Hg)	T_I (°F)	T_F (°F)	Blank abs. (OD)	A_1, abs. (OD)	A_2, abs. (OD)	A_3, abs. (OD)	A_4, abs. (OD)	Sample aliquot (mℓ)	Sample abs. (OD)
1															
2															
3															
4															
5															
6															
7															
8															
9															
10															
11															
12															
13															
14															
15															
16															
17															
18															
19															
20															

STANDARD SOLUTION AND CONTROL SAMPLE ANALYTICAL DATA FORM

Plant _____ Date _____

Analyst _____ Optimum wavelength _____ nm

Blank used as reference? _____

Sample number	Sample, µg	Working solution	Control sample	Measured, absorbance, OD	Calculated absorbance,[a] OD	Absorbance comparison error,[b] %
A1	100	x			-	-
A2	200	x			-	-
A3	300	x			-	-
A4	400	x			-	-
S1	100		x			
S2	200		x			
S3	300		x			
						Avg[c]

$$K_c = 100 \left[\frac{A_1 + 2A_2 + 3A_3 + 4A_4}{A_1^2 + A_2^2 + A_3^2 + A_4^2} \right] = _____.$$

[a] Calculated absorbance: $OD = (\mu g)/K_c$ i.e., S1 calculated absorbance = $100/K_c$.

[b] Absorbance comparison errors:

$$\% = 100 \times \frac{(\text{measured absorbance, OD}) - (\text{calculated absorbance, OD})}{\text{calculated absorbance, OD}}.$$

[c] Average of absolute values.

CALCULATIONS FOR EPA METHOD 7

NOMENCLATURE

The following nomenclature is used in the calculations.

A = absorbance of sample

C = concentration of NO_x as NO_2, dry basis, corrected to standard conditions, mg/dscm (lb/dscf)

F = dilution factor (i.e., 25/5, 25/10, etc.) required only if sample dilution was needed to reduce the absorbance to the range of calibration

K_c = spectrophotometer calibration factor

m = mass of NO_x as NO_2 in gas sample, µg

P_f = final absolute pressure of flask, mm (in.) Hg

P_i = initial absolute pressure of flask, mm (in.) Hg

P_{std} = standard absolute pressure, 760 mm (29.92 in.) Hg

T_f = final absolute temperature of flask, °K (°R)

T_i = initial absolute temperature of flask, °K (°R)

T_{std} = standard absolute temperature, 293°K (528°R)

V_{sc} = sample volume at standard conditions, dry basis, mℓ

V_f = volume of flask and valve, mℓ

V_a = volume of absorbing solution, 25 mℓ

24.06 = molar gas volume at 20°C (68°F) liter

46.005 = molecular weight of nitrogen dioxide

16018463 = conversion factor for lb/dscf to mg/dscm

CALCULATIONS

The following are the equations used to calculate the concentration of nitrogen oxides:

Sample Volume, Dry Basis at Standard Conditions

$$V_{sc} = \frac{T_{std}\, V_f - V_a}{P_{std}} \qquad \frac{P_f}{T_f} - \frac{P_i}{T_i}$$

$$= K_1 \, (V_f - 25 \text{ m}\ell) \, \frac{P_f}{T_f} - \frac{P_i}{T_i} \qquad \text{Equation 7.1}$$

where

$K_1 = 0.3858 \ \frac{^\circ K}{\text{mm Hg}}$ for metric units, or

$K_1 = 17.64 \ \frac{^\circ R}{\text{in. Hg}}$ for English units.

Spectrophotometer Calibration Factor

$$K_c = 100 \ \frac{A_1 + 2A_2 + 3A_3 + 4A_4}{A_1^2 + A_2^2 + A_3^2 + A_4^2} \qquad \text{Equation 7.2}$$

where

A_1 = absorbance of the 100-µg NO_2 standard.

A_2 = absorbance of the 200-µg NO_2 standard.

A_3 = absorbance of the 300-µg NO_2 standard.

A_4 = absorbance of the 400-µg NO_2 standard.

Total µg of NO_2 Per Sample

$$m = 2 \, K_c A F \qquad \text{Equation 7.3}$$

where

2 = 50/25, the aliquot factor (if other than a 25-mℓ aliquot was used for analysis, the corresponding factor must be substituted).

Sample Concentration

$$C = K_2 \quad \frac{m}{V_{sc}} \qquad \text{Equation 7.4}$$

where

$K_2 = 10^3 \quad \frac{mg/m^3}{\mu g/m\ell}$ for metric units, or

$K_2 = 6.243 \times 10^{-5} \quad \frac{lb/scf}{\mu g/m\ell}$ for English units.

When calculated in English units (lb/dscf), the metric unit equivalent (mg/dscm) is determined in this manner:

$$(lb/ft^3)(16.018463) = kg/m^3 \qquad \text{Equation 7.5}$$

therefore:

$$(lb/dscf)(16018463) = mg/dscm \qquad \text{Equation 7.6}$$

Parts per million, dry basis at standard conditions, is determined by the following equation:

$$ppm_{dry} = \frac{(mg/dscm)(24.06)}{46.005} \qquad \text{Equation 7.7}$$

EMISSIONS RATE OF NO_x

$$E_{no_x}\ (lb/hr) = C_{no_x}\ (lb/dscf) \times Q_{std}\ (dscf/hr)$$

where

E_{no_x} = nitrogen oxide emissions rate (lb/hr)

C_{no_x} = concentration of NO_x in stack gases (lb/dscf)

Q_{std} = dry volumetric stack gas flow rate corrected to standard conditions (dscf/hr)

APPENDIX A-4

PROCEDURE, DATA SHEETS, AND CALCULATION SETS

HCL METHOD

DETERMINATION OF HYDROCHLORIC ACID EMISSIONS FROM STATIONARY SOURCES

1. Principle and Applicability

1.1 Principle. A gas sample is extracted from the stack and the hydrochloric acid content is measured using the mercuric nitrate titration method.

1.2 Applicability. This method is applicable for the determination of hydrochloric acid emissions from stationary sources. The upper limit of the method has not been established though samples containing 4000 parts per million hydrochloric acid have been efficiently collected and determined using two midget impingers each containing 15 milliliters of 0.1N sodium hydroxide.

Possible interferences are metal ions (usually Cu, Fe, Ni, Mg, and V) and the sulfite ion. These tend to mask the analysis endpoint. A strong acid type cationic exchange resin has been demonstrated to remove interfering metal ions satisfactorily. When gas streams containing SO_2 in excess of 300 parts per million are sampled, the SO_2 can be effectively removed by treating the sample with 3 percent hydrogen peroxide.

2. Apparatus

2.1 Sampling. The sampling train is shown in Figure 1 and the component parts are discussed below.

2.1.1 Probe. A five-foot, borosilicate glass or Teflon-lined probe is used. The inlet is packed with glass wool to remove particulate matter. The probe must have a heating system to prevent water condensation while sampling.

2.1.2 Midget Impingers. Four midget impingers are connected

in series with leak-free glass connectors. Silicone grease may be used to insure a tight seal.

2.1.3 Glass Wool. Borosilicate or quartz.

2.1.4 Temperature Gauge. Dial thermometer, or equivalent, to measure temperature of gas leaving impinger train to within 1°C (2°F).

2.1.5 Drying Tube. Tube packed with 6-to 16-mesh indicating type silica gel or equivalent to dry the gas sample and to protect the dry gas meter and pump.

2.1.6 Valve. Needle valve, to regulate sample gas flow rate.

2.1.7 Pump. Leak-free diaphragm pump, or equivalent, to pull gas through the train. Install a small surge tank between the pump and the rate meter to eliminate the pulsation effect of the diaphragm pump on the rotameter.

2.1.8 Rate Meter. Rotameter, or equivalent, capable of measuring flow rate to within 4 percent of the selected flow rate of 2 l/min.

2.1.9 Volume Meter. Dry gas meter, sufficiently accurate to measure the sample volume to three decimal places in cubic feet, calibrated at the selected flow rate and conditions actually encountered during sampling, and equipped with a temperature gauge (dial thermometer, or equivalent) capable of measuring temperature to within 3°C (5.4°F).

2.1.10 Barometer. Mercury, aneroid, or other barometer capable of measuring atmospheric pressure to within 2.5 mm Hg (0.1 in Hg).

2.1.11 Vacuum Gauge. At least 760 mm Hg (30 in. Hg) gauge, to be used for leak check of the sampling train.

2.2 Sample Recovery. The following equipment is needed for sample recovery.

2.2.1 Storage Containers. Leak-free 100 ml polyethylene or glass bottles.

2.2.2 Wash Bottle. Polyethylene or glass.

2.3 Analysis. Volumetric glassware should be class A only.

2.3.1 Erlenmeyer Flask. 125 ml.

2.3.2 Graduated Burette. 25 ml.

2.3.3 Volumetric Pipet. 1 ml.

2.3.4 Volumetric Flasks. 100 ml, one for each sample.

2.3.5 Small Funnel. For transferring sample.

2.3.6 Magnetic Stirrer.

2.3.7 Magnetic Stirring Bar.

2.3.8 Dropping Pipet or Dropper.

2.3.9 Filter Paper. Whatman No. 40.

3. Reagents

Unless otherwise indicated, all reagents must conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society. Where such specifications are not available, use the best available grade.

3.1 Sampling.

3.1.1 Water. Triple deionized, distilled to conform to ASTM Specification D 1193-74, Type 3.

3.1.2 Sodium Hydroxide, 0.1 N. Dissolve 4g NaOH in deionized, distilled water and dilute to 1 liter.

3.1.3 Hydrogen Peroxide, 3 Percent. Dilute 30 percent hydrogen peroxide 1:9 (V/V) with deionized, distilled water (15 ml is needed per sample). Prepare fresh daily.

3.2 Sample Recovery.

3.2.1 Sodium Hydroxide, 0. 1N. As in 3.1.2.

3.2.2 Sodium Hydroxide, 0.05 N. Dissolve 2g NaOH in deionized, distilled water and dilute to 1 liter.

3.3 Analysis.

3.3.1 Mercuric Nitrate Solution, 0.01 N. Dissolve 1.63 g mercuric nitrate in deionized, distilled water and dilute to 1 liter. Shake well. Transfer to an amber colored reagent bottle. Standardize this solution according to the procedure in Section 5.5.

3.3.2 Bromophenol-Diphenylcarbazone Indicator. Dissolve 0.005 g bromophenol blue and 0.5 g diphenyl carbazone in 75 ml pure ethanol, then dilute to 100 ml therewith.

3.3.3 0.05 N HNO_3. Dilute 1.6 ml concentrated HNO_3 (69 percent) to 500 ml with distilled, deionized water.

3.3.4 0.0100 N NaCl Solution. Dissolve 0.584 g NaCl in deionized, distilled water and dilute to 1 liter.

3.3.5 Cation Exchange Resin. Rexyn 101 H (Fisher Scientific)[1] is one type that has been used successfully. The resin column is prepared as follows:

A small amount of the fresh resin is poured into a 250 ml beaker containing 100 ml distilled water and allowed to soak for 30 minutes. A 25 ml buret is packed at the bottom end with a small portion of glass wool to prevent plugging the tip with resin particles. The buret is then filled approximately half-full with distilled water, and the wetted resin is poured in until it reaches the 11 ml graduation mark. Care must be taken to maintain

the water level at least one inch above the resin level at all times to prevent resin channelling. The buret is filled to the 0 ml graduation mark with distilled water, and the stopcock is opened to allow the column to drain until the final water level is at the 10 ml graduation mark. This rinsing process is repeated several times and the rinses discarded. A column blank is determined by passing a 10 ml aliquot of distilled water through the column followed by a 10 ml rinse. The blank volume is then adjusted to 100 ml with distilled water and titrated with standard mercuric nitrate. One drop of titrant should be sufficient to reach the endpoint.

4. Procedure

4.1 Sampling

4.1.1 Preparation of Collection Train. Measure 15 ml of 0.1 N sodium hydroxide into each of the first two midget impingers and 15 ml of 3 percent hydrogen peroxide into the third one. Leave the final impinger dry. Assemble the train as in Figure 1. Adjust probe heater to a temperature sufficient to prevent water condensation. Place crushed ice and water around the impingers.

4.1.2 Leak-Check Procedure. A leak-check prior to the sampling run is optional; however, a leak-check after the sampling run is mandatory. The leak check procedure is as follows:

Temporarily attach a suitable (e.g., 0-40 cc/min) rotameter to the outlet of the dry gas meter and place a vacuum gauge at or near the probe inlet. Plug the probe inlet, pull a vacuum of at least 250 mm Hg (10 in. Hg), and note the flow rate as indicated by the rotameter. A leakage rate not in excess of 2 percent of the average sampling rate is acceptable.

Note: Carefully release the probe inlet plug before turning off the pump.

It is suggested (not mandatory) that the pump be leak-checked separately, either prior to or after the sampling run. If done prior to the sampling run, the pump leak-check shall precede the leak-check of the sampling train described immediately above; if done after the sampling run, the pump leak-check shall follow the train leak-check. To leak-check the pump, proceed as follows: Disconnect the drying tube from the probe-impinger assembly. Place a vacuum gauge at the inlet to either the drying tube or the pump, pull a vacuum of 250 mm (10 in.) Hg, plug or pinch off the outlet of the flow meter and then turn off the pump. The vacuum should remain stable for at least 30 seconds.

Other leak-check procedures may be used, subject to the approval of the Administrator, U. S. Environmental Protection Agency.

4.1.3 Sample Collection. Record the initial dry gas meter reading and barometric pressure. To begin sampling, position the tip of the probe at the sampling point, connect the probe to the bubbler, and start the pump. Adjust the sample flow to a constant rate of approximately 2.0 liters/min as indicated by the rotameter. Maintain this constant rate (± 10 percent) during the entire sample run. Take readings (dry gas meter, temperatures at dry gas meter and at impinger outlet and rate meter) at least every 5 minutes. Add more ice during the run to keep the temperature of the gases leaving the last impinger at 20°C (68°F) or less. At the conclusion of each run, turn off the pump, remove probe from the stack, and

record the final readings. Conduct a leak-check as in Section 4.1.2. (This leak-check is mandatory). If a leak is found, void the test run or use procedures acceptable to the Administrator to adjust the sample volume for the leakage. Drain the ice bath, and purge the remaining part of the train by drawing clean ambient air through the system for 15 minutes at the sampling rate.

4.2 Sample Recovery. Disconnect the impingers after purging. Discard the contents of the hydrogen peroxide SO_2 scrubber impinger. Pour the contents of the other impingers containing sodium hydroxide into a leak-free polyethylene or glass bottle for shipment. Rinse the impingers and the connecting tubes with deionized, distilled water, and add the washings to the same storage container. The glass wool probe plug is recovered and placed in a second storage container. The probe is rinse with 25 ml distilled water into a second storage container. The probe is rinsed 25 ml distilled water into a third storage container and marked accordingly. Seal, mark the fluid level, and identify all containers.

4.3 Sample Analysis. Note level of liquid in each container, and confirm whether any sample was lost during shipment; note this on analytical data sheet. If a noticeable amount of leakage has occurred, either void the sample or use methods, subject to the approval of the Administrator, to correct the final results.

Using a small funnel, the contents of each sample container are quantitatively transferred to separate 100 ml volumetric flasks. The probe wash is filtered through Whatman Number 40 paper into a flask. The probe plug is rinsed three times with water and likewise filtered into another flask. Each flask is diluted to the mark with deionized, distilled water.

An aliquot of one out of every five samples is passed through a cationic exchange resin (prepared as in Section 3.3.5) and compared to that of untreated samples to see if interfering metal ions are present. If values differ by more than 1 percent, an aliquot of all samples should be passed through the resin prior to the analysis. To oxidize any SO_2 present, add and mix 2 ml 3 percent hydrogen peroxide solution per 100 ml sample.

Before analysis, the pH of the sample must be adjusted to 3.2 - 3.4. Pour 25 ml of the 100 ml sample into a 125 ml Erlenmeyer flask and add 5 drops of bromophenol/diphenyl carbazone while stirring the sample continuously using a magnetic stirring bar. If the sample is initially alkaline, it will turn blue or red in color after addition of the indicator. In this case, add 0.1 N nitric acid dropwise until the sample just turns yellow in color. Then add an additional 1.0 ml of 0.05 N nitric acid to bring the pH to 3.2 - 3.4. If the sample is initially acidic, the solution will turn yellow after addition of the indicator. In this case, add 0.1 N sodium hydroxide dropwise until the sample turns blue in color. Then add 0.05 N nitric acid dropwise until the sample just turns yellow. Add an additional 1.0 ml of 0.05 N nitric acid to bring the sample to the proper pH of 3.2 - 3.4. After the pH is adjusted, titrate the sample with standard 0.01 N mercuric nitrate until the first appearance of a violet color which persists for several minutes with continuous stirring. Repeat and average the tiration volumes. Run a blank with each series of samples.

5. Calibration

5.1 Metering System. Before its initial use and after each field test series, leak check the metering system as described in Method 6,

Section 5.1.

5.2 Thermometers. Calibrate against mercury-in-glass thermometer.

5.3 Rotameter. The rotameter need not be calibrated but should be kept clean according to manufacturer's instructions.

5.4 Barometer. Calibrate against mercury barometer.

5.5 Mercuric Nitrate Solution. Standardize the mercuric nitrate solution by titrating against triplicate aliquots of 0.0100 N standard NaCl solution.

6. Calculations

Carry out calculations, retaining at least one extra decimal figure beyond that of the acquired data. Round off figures after the final calculation. Other forms of the equations may be used as long as they give equivalent results.

6.1 Nomenclature

C_{HCl} = Concentration of hydrogen chloride, dry basis corrected to standard conditions, mg/dscm (lb/dscf).

N = Normality of mercuric nitrate titrant, milliequivalents/ml.

P_{bar} = Barometric pressure at the exit orifice of the dry gas meter, mm Hg (in. Hg).

P_{std} = Standard absolute pressure, 760 mm Hg (29.92 in. Hg).

T_m = Average dry gas meter absolute temperature, °K (°R).

T_{std} = Standard absolute temperature, 293°K (528°R).

V_a = Volume of sample aliquot titrated, ml.

V_m = Dry gas volume as measured by the dry gas meter, dcm (dcf).

$V_{m(std)}$ = Dry gas volume measured by the dry gas meter, corrected to standard conditions, dscm (dscf).

V_{soln} = Total volume of solution in which the hydrochloric acid sample is contained, 100 ml.

V_t = Volume of mercuric nitrate titrant used for the sample, ml (average of replicate titrations).

V_{tm} = Volume of mercuric nitrate titrant used for the blank, ml.

Y = Dry gas meter calibration factor.

70.90 = Equivalent weight of hydrochloric acid.

6.2 Dry sample gas volume, corrected to standard conditions.

$$V_{m(std)} = V_m Y \left(\frac{T_{std}}{T_m}\right)\left(\frac{P_{bar}}{P_{std}}\right) = K_1 Y \frac{V_m P_{bar}}{T_m}$$

Equation 6-1

Where:

K_1 = 0.3858 °K/mm Hg for metric units.

= 17.64 °R/in. Hg for English units.

6.3 Hydrochloric acid concentration.

$$C_{HCl} = K_2 \frac{(V_t - V_{tm})\left(\frac{V_{soln}}{V_a}\right) N}{V_{m(std)}}$$

Equation 6-2

Where:

K_2 = 70.90 mg/meg. for metric units

= 1.563×10^{-4} lb/meg. for English units.

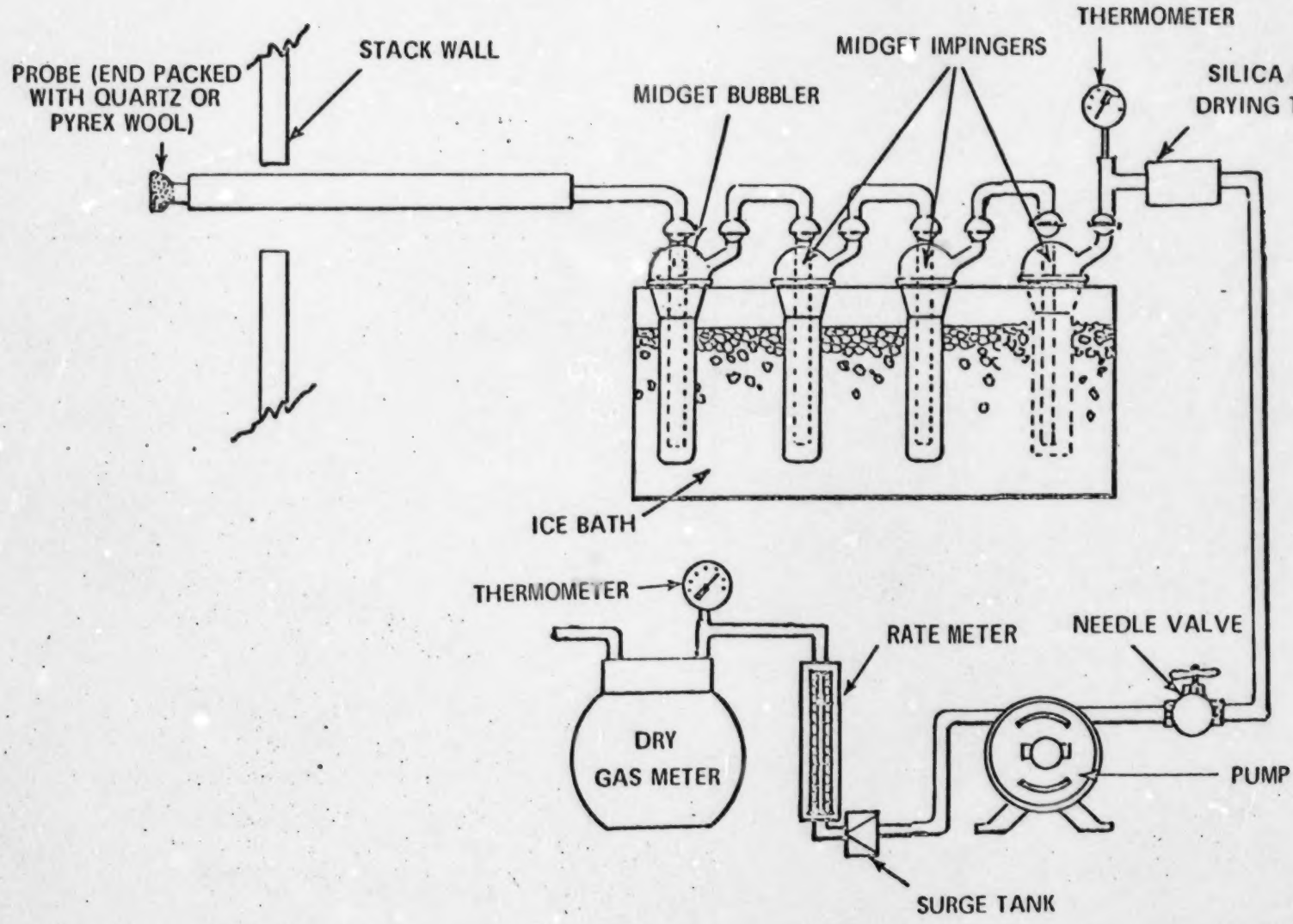
7. Bibliography

1. Cheney, J., C. Fortune, and R. Rollins. Report on the Development and Evaluation of Stationary Source Hydrochloric Acid Emissions Measurement Methodology. U. S. Environmental Protection Agency, Stationary Source Emissions Research Branch. Research Triangle Park, N. C. December, 1976.

2. Rom, J. J. Maintenance, Calibration, and Operation of Isokinetic Source-Sampling Equipment. Office of Air Programs, Environmental Protection Agency. Research Triangle Park, N. C. APTD-0576. March, 1972.

3. Annual Book of ASTM Standards. Part 31; Water, Atmospheric Analysis. American Society for Testing and Materials. Philadelphia, Pa. 1974. pp. 40-42.

[1] Mention of trade names or specific products does not constitute endorsement by the Environmental Protection Agency.



Hydrochloric acid emissions sampling train.

Systech Corporation
245 North Valley Road
Xenia, Ohio 45385
(513) 372-8077
Dayton Number: (513) 429-2533

FIELD SAMPLING DATA FORM FOR EPA DRAFT PROCEDURE DETERMINATION OF HYDROCHLORIC ACID EMISSIONS FROM STATIONARY SOURCES

Operator _____

Project number _____ Sample number _____

Plant name _____ Barometric pressure (in. Hg) _____

City _____ Ambient temperature (°F) _____

Date _____ Probe length (ft) _____

Sample point location _____ Probe heater setting _____

Meter box number _____ Probe material _____

Initial leak check _____ Sample purge time (min) _____

Final leak check _____ Remarks _____

Sampling time (min)	Clock time (24 hr)	Sample flow rate setting (ℓ/min)	Sample volume metered (ft^3)	Dry gas meter temp. (°F)
0				
5				
10				
15				
20				

Moisture data		
Combined impinger & drying tube wt (g) (posttest)	Combined impinger & drying tube wt (g) (pretest)	Weight gain (g)

HYDROCHLORIC ACID ANALYTICAL DATA

Plant _____ Date _____

Sample location _____ Analyst _____

Volume and normality of barium perchlorate

1 _____ ml _____ N

2 _____ ml _____ N _____ N, avg

3 _____ ml _____ N

Sample number	Sample identification number	Total sample volume (V_{soln}), ml	Sample aliquot volume (V_a)[a], ml	Volume of titrant (V_t)[b], ml		
				1st titration	2nd titration	Average
1						
2						
3						
4						
5						
6						
Blank		N/A				V_{th} =

[a] Volume for the blank must be the same as that of the sample aliquot.

[b] $\frac{\text{1st titration}}{\text{2nd titration}}$ = 0.99 to 1.01 or $|\text{1st titration} - \text{2nd titration}| \leq 0.2$ ml.

Signature of analyst _____

Signature of reviewer or supervisor _____

Systech Corporation
345 North Valley Road
Xenia, Ohio 45385
(513) 372-8077
Dayton Number (513) 429-2533

EMISSIONS RATE OF HCl

$$E_{HCl}\ (lb/hr) = C_{HCl}\ (lb/dscf) \times Q_{std}\ (dscf/hr)$$

where

E_{HCl} = Hydrochloric acid emissions rate (lb/hr)

C_{HCl} = Concentration of HCl in stack gases (lb/hr)

Q_{std} = Dry volumetric stack gas flow rate corrected to standard conditions (dscf/hr)

APPENDIX A-5

DATA SHEETS AND CALCULATION SETS

MODIFIED METHOD 8

PARTICULATE FIELD DATA + SO_2

PLANT _____ AMBIENT TEMPERATURE _____ NOZZLE DIAMETER, in. _____ STACK NO. _____

DATE _____ BAROMETRIC PRESSURE _____ STACK DIAMETER, in. _____ RUN NO. _____

LOCATION _____ ASSUMED MOISTURE, % _____ METER $\Delta H @$ _____ FUEL _____

OPERATOR _____ PROBE LENGTH, in. _____ C FACTOR _____ BOILER SIZE _____

SAMPLING TIME/POINT _____ LEAK CHECK _____ PROCESS WEIGHT RATE _____

TIME	TRAVERSE POINT		VELOCITY HEAD Δ Ps	STATIC PRESSURE (IN. H_2O)	PUMP VACUUM IN Hg GUAGE	PRESSURE DIFFERENTIAL ACROSS ORIFICE METER (ΔH) IN. H_2O ACTUAL	DRY GAS SAMPLE VOL. (Vm) FT^3	TEMPERATURES °F					
START /STOP	NO.	LOCATION IN.						STACK (T_s)	PROBE	OVEN	IMP OUT	IMP IN	METER OUT (Tm)

ORSAT MEASUREMENT

	TIME	CO_2	O_2	CO	N_2
1					
2					
3					
4					
5					
6					

COMMENTS:

_____

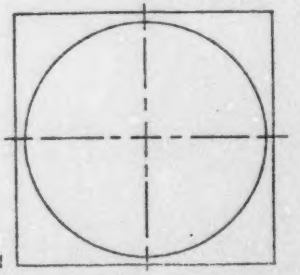
_____

_____

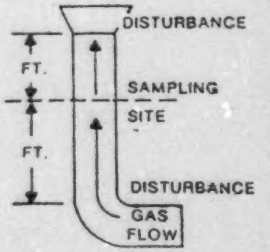
_____

_____

SCHEMATIC OF STACK



CROSS SECTION



Reproduced from best available copy.

SULFUR DIOXIDE ANALYTICAL DATA

Plant _____ Date _____

Sample location _____ Analyst _____

Volume and normality of barium perchlorate

1 _____ ml _____ N

2 _____ ml _____ N _____ N, avg

3 _____ ml _____ N

Sample number	Sample identification number	Total sample volume (V_{soln}), ml	Sample aliquot volume (V_a),[a] ml	Volume of titrant (V_t),[b] ml		
				1st titration	2nd titration	Average
1						
2						
3						
4						
5						
6						
Blank		N/A				V_{tb} =

[a] Volume for the blank must be the same as that of the sample aliquot.

[b] $\frac{\text{1st titration}}{\text{2nd titration}}$ = 0.99 to 1.01 or $\left|\text{1st titration} - \text{2nd titration}\right| \leq 0.2$ ml.

Signature of analyst _____

Signature of reviewer or supervisor _____

Systech Corporation
245 North Valley Road
Xenia, Ohio 45385
(513) 372-8077
Dayton Number: (513) 429-2533

Systems Technology Corporation
245 North Valley Road
Xenia, Ohio 45385
(513) 372-8077
Dayton Number: (513) 429-2533

EPA Q.A. SO_2 REFERENCE STANDARDS ANALYTICAL DATA FORM

Plant _____ Data analyzed _____

Analyst _____ $N_{Ba(ClO_4)_2}$ _____

Aliquot size (V_A) _____ mℓ

Volume metered corrected to standard conditions (Vm[std]) _____ DSCM
× 35.314667 = _____ DSCF

Volume of solution (V soln) _____ mℓ

Titration of blank _____ mℓ $Ba(ClO_4)_2$ (must be <0.5 mℓ)

Control sample number	Time of analysis, 24 hr	Titrant volume, [a] mℓ		
		1st	2nd	Avg

[a] Two titrant volumes must agree within 0.2 mℓ.

(Accuracy of control samples must be within ± 5 percent.)

Does value agree? _____ yes _____ no

_____ Signature of analyst

_____ Signature of reviewer

Systems Technology Corporation
245 North Valley Road
Xenia, Ohio 45385
(513) 372-8077
Dayton Number: (513) 429-2533

EPA Method 6 - SO_2 Computer Worksheet

Client _____ Location _____ SYSTECH P.N. _____

	Sample No.	Vm DCF	Pbar in. Hg	Tm °R	Vt mℓ	Vtb mℓ	N meq./mℓ	Va mℓ	V soln mℓ	Y	(If sample vol. loss encountered) V soln (I)	V soln (F)
1												
2												
3												
4												
5												
6												
7												
8												
9												
10												
11												
12												
13												
14												
15												
16												
17												
18												
19												
20												

CALCULATIONS FOR EPA METHOD 6

NOMENCLATURE

The following nomenclature is used in the calculations.

B_{ws} = water vapor in gas stream, proportion by volume

C_{SO_2} = concentration of sulfur dioxide, dry basis corrected to standard conditions, g/dscm (lb/dscf)

M_w = molecular weight of water, 18.0 g/g-mole (18.0 lb/lb-mole)

N = normality of barium perchlorate titrant, meq/mℓ

P_{bar} = barometric pressure at the exit orifice of the dry gas meter, mm (in.) Hg

P_{std} = standard absolute pressure, 760 mm (29.92 in.) Hg

R = ideal gas constant, 0.06236 (mm Hg)(m^3)/(g-mole)(°K) for metric units and 21.85 (in. Hg)(ft^3)/(lb-mole)(°R) for English units

T_m = dry gas meter average absolute temperature, °K (°R)

T_{std} = standard absolute temperature, 293°K (528°R)

V_a = volume of sample aliquot titrated, mℓ

V_m = dry gas volume measured by dry gas meter, dcm (dcf)

$V_{m(std)}$ = dry gas volume measured by dry gas meter, corrected to standard conditions, dscm (dscf)

V_{soln} = total volume of solution in which the sulfur dioxide sample is contained, 100 mℓ

V_t = volume of barium perchlorate titrant used for the sample (average of replicate titrations), mℓ

V_{tb} = volume of barium perchlorate titrant used for the blank, mℓ

$V_{wsg(std)}$ = volume of water vapor collected in impingers and silica gel, scm (scf)

W_f = final combined weight of impingers and silica gel, grams

W_i = initial combined weight of impingers and silica gel, grams

Y = dry gas meter calibration factor

24.06 = molar gas volume at 20°C (68°F), liters

32.03 = equivalent weight of sulfur dioxide

64.06 = molecular weight of sulfur dioxide

16018463 = conversion factor for lb/dscf to mg/dscm

CALCULATIONS

The following formulas for calculating the concentration of sulfur dioxide were used:

Dry Sample Gas Volume, Corrected to Standard Conditions

$$V_{m\ (std)} = V_m Y \frac{T_{std}\ P_{bar}}{T_m\ P_{std}} = K_1 Y \frac{V_m P_{bar}}{T_m} \qquad \text{Equation 6.1}$$

where

K_1 = 0.3858 °K/mm Hg for metric units, or

= 17.64 °R/in. Hg for English units.

Sulfur Dioxide Concentration

$$C_{SO_2} = K_2 \frac{V_t - V_{tb}\ \ N \frac{V_{soln}}{V_a}}{V_{m(std)}} \qquad \text{Equation 6.2}$$

where

K_2 = 32.03 mg/meq for metric units, or

= 7.061×10^{-5} lb/meq for English units.

When calculated in English units (lb/dscf), the metric unit equivalent (mg/dscm) is determined in this manner:

$(lb/ft^3)\ (16.018463)^* = (kg/m^3)$ Equation 6.3

---

* C.R.C. Handbook of Chemistry and Physics, 53rd Ed., 1972-73. Pg. F-272.

Therefore:

$$(lb/dscf)(16018463) = (mg/dscm) \qquad \text{Equation 6.4}$$

Parts per million, dry basis at standard conditions, is determined by the following equation:

$$ppm_{dry} = \frac{(mg/dscm)(24.06)}{(64.06)} \qquad \text{Equation 6.5}$$

Water Vapor Collected in Impingers and Silica Gel

$$V_{wsg(std)} = \frac{W_f - W_i \quad RT_{std}}{P_{std} \; M_w} = K_2 \quad W_f - W_i \qquad \text{Equation 6.6}$$

where

$K_2 = 0.001335\ m^3/g$ for metric units, or

$= 0.04715\ ft^3/g$ for English units.

Water Vapor in Gas Stream, Proportion by Volume

$$B_{ws} = \frac{V_{wsg(std)}}{V_{wsg(std)} + V_{m(std)}} \qquad \text{Equation 6.7}$$

PPM SO_2, Wet Basis

$$ppm_{wet} = ppm_{dry} \quad 1 - B_{ws} \qquad \text{Equation 6.8}$$

EMISSIONS RATE OF SO_2

$$E_{SO_2}\ (lb/hr) = C_{SO_2}\ (lb/dscf) \times Q_{std}\ (dscf/hr)$$

where

E_{SO_2} = Sulfur dioxide emission rate (lb/hr)

C_{SO_2} = Concentration of SO_2 in stack gases (lb/dscf)

Q_{std} = Dry volumetric stack gas flow rate corrected to standard conditions

APPENDIX A-6

DATA SHEETS AND CALCULATION SETS

O_2, CO, AND CO_2

INSTRUMENT CALIBRATION DATA

PROJECT: _____ DATE: _____

LOCATION: _____ OPERATOR: _____

	SO_x		NO_x		O_2		CO_2		CO				COMMENTS
Chanel													
Std. Conc.													
Std. Span													
Date/Time	Zero	Span	Zero	Span	Zero	Span	Zero	Span	Zero	Span	Zero	Span	

Oxygen, carbon monoxide, and carbon dioxide concentrations in the stack gas will be determined by averaging the percent of scale readouts at each 5-min time line on the strip chart over the 4-hr test period. This average value will be read off the calibration curve for that instrument and reported as percent O_2, percent CO_2, or ppm CO. In the event of instrument drift or deviation from proper calibration, the following calculation will be applied to average out the effect of this deviation.

$$C_s = \frac{C_{std}\,[(R_s - Z_{fc}) + R_s]/2}{[R_{ic} + (R_{fc} - Z_{fc})]/2}$$

$$C_s = \frac{C_{std}\,[(R_s - Z_{fc}) + R_s]}{R_{ic} + (R_{fc} - Z_{fc})}$$

where

C_{std} = concentration of standard (ppm or percent)

R_{ic} = reading initial calibration (percent of scale)

R_{fc} = reading final calibration (percent of scale)

Z_{fc} = zero drift final calibration (percent of scale)

R_s = reading of sampling - hourly average (percent of scale)

C_s = concentration of sample (ppm or percent)

APPENDIX A-7

DATA SHEETS AND CALCULATION SETS

METHOD 23

METHOD 23

Field Sampling Data

Date _____ Sample I.D. _____

Plant _____ Bag Leak Check _____

Technician _____

Start time _____ Flow rate _____

Stop time _____ Flow rate _____

POHC Analytical Date

Date of Sampling _____

Plant Location _____

POHCs Analyzed _____

Date of Analysis _____

Analyst Responsible _____

Reviewed by _____

Date of Review _____

Column flow rate (ml/min) _____

Oven Conditions _____

Full scale (mv) _____

Electrometer set (A/mv) _____

Recorder speed (in/min) _____

Sample size (ml) _____

Comments _____

_____

_____

_____

POHC ANALYTICAL DATA

Run No.	Time	Sample/Standard	POHC component	Retention time	Attentuation	Peak Area	Blank Area	Corrected Peak Area	Concentration (ppm) (C_c)

FOHC ANALYTICAL DATA

Run No.	Time	Sample/Standard	FOHC component	C_c (pg. 2)	P_r	T_l	P_l	T_r	$(1-S_{wb})$	Sample Concentrations (C_s)

$$C_s = \frac{C_c P_r T_l}{P_l T_r (1-S_{wb})}$$

Where:

C_s = Sample Concentration (ppm)

C_c = Indicated Concentration (ppm)

P_r = Reference Pressure (mm Hg)

T_l = Sample loop temperature (^oK)

P_l = Pressure (mm Hg)

T_r = Reference sample loop temp. (^oK

S_{wb} = Sample water vapor fraction (from Method 5 data)

EMISSIONS RATE OF POHC

$$\text{POHC lb/hr} = C_s \text{ ppm} \times \frac{MW_{POHC}}{24.056 \text{ ℓ/mole}} \times \frac{2.205 \times 10^{-6} \text{ lb/mg}}{35.314 \text{ ft}^3/\text{m}^3} \times Q_{std}$$

DESTRUCTION AND REMOVAL EFFICIENCY

$$DRE = \frac{POHC_{in} \text{ (lb/hr)} - POHC_{out} \text{ (lb/hr)}}{POHC_{in} \text{ (lb/hr)}} \times 100$$

where

C_s ppm = Sample concentration

MW_{POHC} = Molecular weight of POHC

Q_{std} = Stack flow rate (dscf/hr)

APPENDIX A-8

LIQUID FUEL FEED RATE

ESP DUST ACCUMULATION RATE

LIQUID FUEL FEED RATE

Plant _____

Operator _____

Time	Liquid level in truck	Truck Vol. (from curve)	Nozzle Pressure	Flow rate (from curve)	Air Pressure	Comments
	inches	gallons	psi	gpm	psi	

LIQUID FUEL FEED CALCULATIONS

FEED RATE (TRUCK LEVEL METHOD)

$$Lf_r = \frac{V_i - V_f}{t_i - t_f}$$

where

Lf_r = Liquid fuel feed rate (gal/min)

t_i = Time of initial measurement

t_f = Time of final measurement

V_i = Volume of truck at t_i (gal)

V_f = Volume of truck at t_f (gal)

FEED RATE (NOZZLE PRESSURE METHOD)

Lf_r is read from a curve which will be generated during the drum calibration procedure outlined in Appendix B. This curve will be developed prior to the test burn and can be verified or updated at any time during the test burn.

HEAT INPUT

$$H_{Lf} = HHV_{Lf} \times Lf_r \times SG_{Lf}$$

where

H_{Lf} = Heat input rate of liquid fuel (Btu/hr)

HHV_{Lf} = High heating value of liquid fuel (Btu/lb)

Lf_r = Feed rate of liquid fuel (gal/min)

SG_{Lf} = Specific gravity of liquid fuel (lb/gal)

PERCENT HEAT INPUT

$$\text{percent } H_{Lf} = \frac{H_{Lf}}{H_{Lf} + H_c} \times 100$$

where

Percent H_{Lf} = Percent of total heat input derived from liquid fuel

H_{Lf} = Heat input rate of liquid fuel (Btu/hr)

H_c = Heat input rate of coal (Btu/hr)

E.S.P. DUST ACCUMULATION RATE

PROJECT: _____

DATE: _____

	TIME	TRUCK NO.	GROSS WT.	TARE WT.	NET WT.	LOC.
1.						
2.						
3.						
4.						
5.						
6.						
7.						
8.						
9.						
10.						
11.						
12.						
13.						
14.						
15.						
16.						
17.						
18.						
19.						
20.						
21.						
22.						

APPENDIX B

QUALITY ASSURANCE

QUALITY ASSURANCE

OVERVIEW

Whenever possible, the final results from this test will be accompanied by a statement of data quality. This data qualification may be in the form of precision and accuracy statements, lower and/or upper detection limits of the method, or merely a warning to the reader that a particular set of data was acquired while the process was somewhat unstable.

The following section outlines some of the procedures that SYSTECH will follow to ensure that the data resulting from this test will be of known high quality or identified as questionable if circumstances warrant. Figure B-1 summarizes the major areas of field quality assurance that SYSTECH will address.

CONFIGURATION CONTROL LOG

A detailed log will be maintained which will illustrate the configuration of all pertinent aspects of the field test. This log will be initiated during the pretest burn period and will be expanded and updated during the equipment set up, pretest dry run, and actual test periods. The intent of this log is to reflect as accurately as possible the configuration and functional status of the liquid fuel injection system and all test measurement systems at any given time during the test sequence. This information will be reviewed daily for possible impact on the final test results as a part of the data validation procedures.

SAMPLE CONTROL

To ensure the credibility of the samples acquired in the field, the following procedures will be followed.

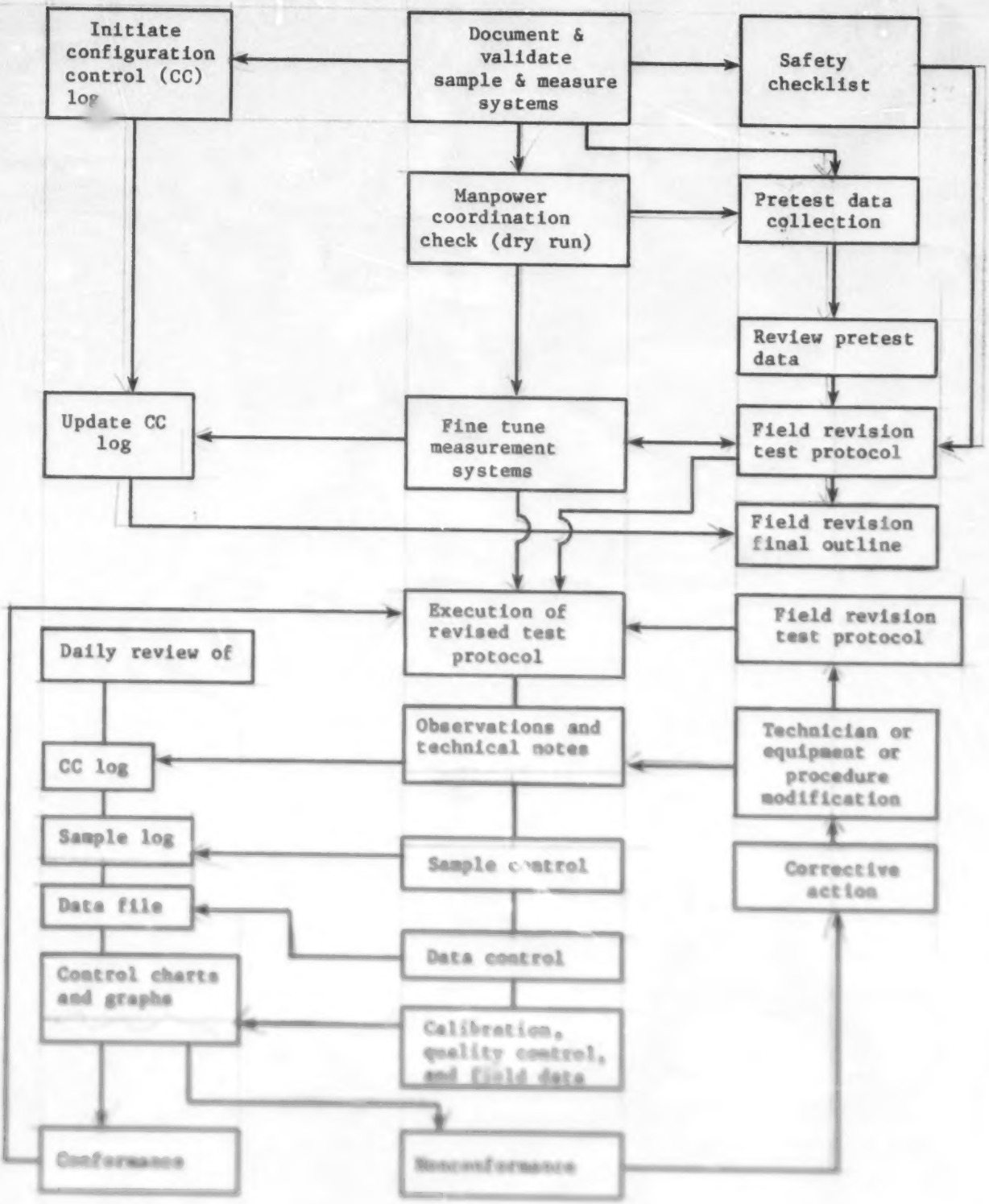


Figure D-1. Field quality assurance overview.

Samples will be identified by the responsible technician as soon as possible after sampling or sample recovery. The sample will be identified by filling out and affixing the preprinted label illustrated in Figure B-2.

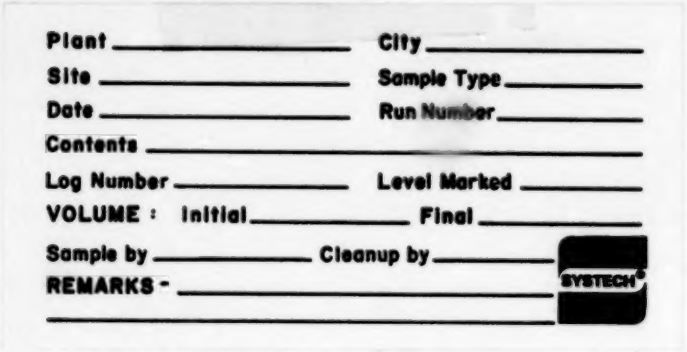


Figure B-2. Sample identification label.

The labeled sample containers will be entered into the sample log (Figure B-3) and assigned a log number by the field chemist in charge as soon as possible after sampling, not later than the end of the test day. The sample log will be checked against the expected number and type of samples indicated on the sample inventory sheet (Figure B-4). The logged samples will be placed in a limited access storage cabinet in the SYSTECH mobile laboratory until the end of the test sequence. The samples will then be transported by SYSTECH personnel to the analytical laboratories where they will be signed over to the responsible laboratory person.

Approximately four hourly coal, liquid fuel, ESP dust, and clinker samples will be composited to create one analytical sample of each sample type for each test period. A preliminary review of the stack test data sheets

ENVIRONMENTAL ENGINEERING SPECIALISTS
SYSTEMS TECHNOLOGY CORPORATION
245 N. Valley Rd. • Xenia, Ohio 45385 • 513/372-8077
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DATE	TIME	ID NUMBER	SAMPLE DESCRIPTION	LOGGED BY	LOCATION - COMMENTS OBSERVATIONS - TECHNICAL NOTES

Figure B-3. Sample log.

Sample description	Number of samples per day
M-5 filter	2 each
M-5 probe rinse	2 each
M-5 impinger sol.	2 each
M-7 absorbing sol.	6 each
M-25 tank	4 each
M-25 trap	4 each
HCl filter	2 each
HCl probe rinse	2 each
HCl impingers	2 each
Coal/coke	8 or more
Liquid fuel	8 or more
ESP dust	8 or more
Clinker	8 or more

Figure B-4. Sample inventory.

will indicate which hourly samples should be composited to best represent any given test period. Approximately one fourth of each hourly sample will be separated in such a way as to maintain the representativeness of the sample. The same weight or volume from each indicated hourly sample will be combined and mixed to form the composite analytical sample. The remaining three fourths of each hourly sample will be maintained as a record sample for future reference.

DATA CONTROL

Field data sheets will be filled out by the responsible technician before being logged in by the field chemist at the conclusion of each test day. The data sheets will be checked for completeness, and three copies of each data sheet will be generated. The original form will go into the master data file under the direct supervision of the project manager. The three copies will be considered as working copies to be used for subsequent calculations, laboratory coordination, and technical project reviews.

All calculations and data reductions will be spot checked for accuracy. All preliminary data as well as final results will undergo critical review for irregularities and will be cross checked against available laboratory quality control and calibration data, and observations of unusual circumstances which are noted in the configuration control log.

CALIBRATIONS

It is beyond the scope of this document to list the hundreds of pieces of sampling and analytical apparatus used in this test that will undergo calibration or will be verified to be within manufacturers' specifications. However, there are a few measurement system calibrations which should be detailed because of potential impact on data or because of unorthodox calibration techniques.

Coal Belt Scale

The coal feed rate will be monitored by taking hourly readings from a weightometer (#WLSV10188) belt scale manufactured by the Merrick Scale Manufacturing Co. This scale will be calibrated prior to the test sequence by Marquette personnel. The calibration procedure consists of passing a length of heavy log chain of known weight across the belt scale. The readout mechanism is then adjusted and the procedure repeated until the correct weight is displayed.

Liquid Fuel Delivery Rate

The feed rate of the liquid fuel through the nozzle will be established by determining the time required to drain a 55-gal drum. This drum will be plumbed into the liquid fuel feed system as illustrated in Figure 2. This plumbing configuration will allow the drum to be filled and emptied by repositioning two 3-way valves without disturbing normal fuel feed. The drum will be fitted with a check valve near the top to avoid overfilling. The volume of the drum filled to the level of the check valve will be determined prior to testing. Once filled, the drum can be substituted for the feed truck in the feed system, the time required to drain the drum determined, and the feed rate calculated. Since this procedure does not disturb normal fuel feed, it can be performed any time there is change in the feed system or an observed change in nozzle pressure.

Oxygen, Carbon Monoxide, and Carbon Dioxide Sampling System

The continuous monitors for O_2, CO, and CO_2 will be calibrated during the morning and evening of each test day with commercially prepared gas standards. These standards are certified by Matheson Incorporated to be within 2 percent of the analyzed value. The O_2 monitor will be subjected to a zero-span (10 percent O_2) calibration. The CO monitor will receive a 4-point calibration (zero, 200, 400, and 800 ppm CO). These calibration gases will be introduced directly into the analytical instruments. The CO_2 monitor

will also be calibrated with four calibration gases (zero, 15, 30, and 50 percent CO_2).

The overall integrity of the sampling system will be validated by introducing one of the gas standards via a 50-ℓ bag into a 3-way valve with a T-fitting on the sample line just outside the stack. The total system response time and possible system biases will be determined with this procedure.

LABORATORY QUALITY CONTROL DATA

It is beyond the scope of this document to present a detailed list of all the analytical quality control data which will be available to qualify the data from this test. The laboratories designated to perform the majority of the analyses do so on a routine basis and have routine quality control data which will be available for review. Nonroutine samples which will require specialized quality control attention include the following: M-23 and GC/FID, chloride and sulfur determinations on ESP dust and clinker, and hydrochloric acid determination.

Method 23 outlines appropriate quality control procedures which will be adhered to as closely as possible. In addition, all samples will be bracketed by standards to ensure the instrument response is consistent. The chloride and sulfur determinations will be run in duplicate to ensure reproducible results. The hydrochloric acid procedure outlines methods to verify the presence of interferences and to eliminate them if present. These samples will also be run in duplicate.